Validation Qualifiers in obtabase

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CETIFICATION

SDG No:

JC33375

Laboratory:

Accutest, New Jersey

Site:

BMS, Building 5 Area, PR

Matrix:

Groundwater

Humacao, PR

SUMMARY:

Groundwater samples (Table 1) were collected on the BMSMC facility – Building 5 Area. The BMSMC facility is located in Humacao, PR. Samples were taken December 6, 2016 and were analyzed in Accutest Laboratory of Dayton, New Jersey for the parameters shown in Table 1. The results were reported under SDG No.: JC33375. Results were validated using the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. Individual data review worksheets are enclosed for each target analyte group. The data sample summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE DESCRIPTION	MATRIX	ANALYSIS PERFORMED
JC33375-1	OSMW-3S	Groundwater	SVOCs: PAHs + 1,4-Dioxane (SIM); Pesticides; Inorganics; Methane
JC33375-2	OSMW-4S	Groundwater	SVOCs: PAHs + 1,4-Dioxane (SIM); Pesticides; Inorganics; Methane

Reviewer Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

January 14, 2017

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Mendez

IC # 188

Report of Analysis

Page 1 of 1

Client Sample ID: OSMW-3S JC33375-1

Lab Sample ID: Matrix:

AQ - Ground Water

SW846 8270D SW846 3510C BMSMC, Building 5 Area, PR Date Sampled: 12/06/16 Date Received: 12/09/16

Percent Solids: n/a

Project:

File ID Prep Date Prep Batch **Analytical Batch** DF Analyzed By Run #1 P109786.D 1 12/14/16 RL 12/13/16 OP99167 EP4874

Run #2

Method:

Initial Volume Final Volume Run #1 1000 ml 1.0 ml

Run #2

CAS No. Compound Result RL MDL Units Q

100-52-7 0.29 Benzaldehyde ND 5.0 ug/l

CAS No. Surrogate Recoveries Run#1 Run#2 Limits

4165-60-0 Nitrobenzene-d5 70% 32-128% 321-60-8 2-Fluorobiphenyl 81% 35-119% 10-126% 1718-51-0 Terphenyl-d14 94%





MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



SGS Accutest LabLink@939893 10:02 27-Dec-2016

Report of Analysis

By

SG

Page 1 of 1

Client Sample ID: OSMW-3S Lab Sample ID: JC33375-1

File ID

3P57222.D

Matrix:

AQ - Ground Water

DF

1

Date Sampled: 12/06/16 Date Received: 12/09/16

Method:

SW846 8270D BY SIM SW846 3510C

Analyzed

12/14/16

Percent Solids: n/a

Q

Prep Date

12/13/16

Project:

BMSMC, Building 5 Area, PR

Prep Batch OP99167A

Analytical Batch E3P2653

Run #1 Run #2

> Initial Volume Final Volume 1000 ml 1.0 ml

Run #1 Run #2

CAS No. Compound Result RL MDL Units 56-55-3 Benzo(a)anthracene ND 0.050 0.023 ug/l 91-20-3 Naphthalene ND 0.10 0.029 ug/l 123-91-1 1,4-Dioxane 1.73 0.10 0.049 ug/l

CAS No. Surrogate Recoveries Run#1 Run#2 Limits

4165-60-0 Nitrobenzene-d5 71% 24-125% 321-60-8 2-Fluorobiphenyl 74% 19-127% 1718-51-0 Terphenyl-d14 68% 10-119%



ND = Not detected

MDL = Method Detection Limit

J = Indicates an estimated value

RL = Reporting Limit

B = Indicates analyte found in associated method blank

E = Indicates value exceeds calibration range



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Report of Analysis

Page 1 of 1

Client Sample ID: OSMW-3S Lab Sample ID: JC33375-1

Matrix:

AQ - Ground Water RSK-175

BMSMC, Building 5 Area, PR

Date Sampled: 12/06/16

Date Received: 12/09/16

Percent Solids: n/a

File ID DF Analyzed By Prep Date Prep Batch Analytical Batch Run #1 AA56399.D 1 12/16/16 LM n/a n/a **GAA1095**

Run #2

Method:

Project:

CAS No. Compound Result RL MDL Units Q

74-82-8 Methane 3.2 0.11 0.036 ug/l



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Page 1 of 1

SGS Accutest LabLink@939893 10:02 27-Dec-2016

Report of Analysis

Client Sample ID: OSMW-3S

Lab Sample ID: JC33375-1 Matrix: AQ - Ground Water

Method: SW846 8081B SW846 3510C

Project: BMSMC, Building 5 Area, PR Date Sampled: 12/06/16 Date Received: 12/09/16

Percent Solids: n/a

File ID DF Prep Date Prep Batch Analytical Batch Analyzed By Run #1 1G130505.D 12/14/16 KD 12/13/16 OP99172 G1G4171

Run #2

Initial Volume Final Volume Run #1 960 ml 10.0 ml

Run #2

CAS No. Compound Result RL MDL Units Q

60-57-1 Dieldrin ND 0.010 0.0038 ug/l

CAS No. Surrogate Recoveries Run#1 Run#2 Limits

877-09-8 Tetrachloro-m-xylene 97% 26-132% 877-09-8 Tetrachloro-m-xylene 90% 26-132% 2051-24-3 Decachlorobiphenyl 60% 10-118% 60% 2051-24-3 Decachlorobiphenyl 10-118%



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Report of Analysis

Page 1 of 1

Client Sample ID: OSMW-3S

Lab Sample ID:

JC33375-1 AQ - Ground Water Date Sampled: 12/06/16

Date Received: 12/09/16

Project:

Matrix:

BMSMC, Building 5 Area, PR

Percent Solids: n/a

Total Metals Analysis

Analyte	Result	RL	MDL	Units	DF	Ргер	Analyzed By	Method	Prep Method
Iron Manganese	3020 379	100 15	12 0.39	ug/l ug/l				SW846 6010C ¹ SW846 6010C ¹	

(1) Instrument QC Batch: MA40966

(2) Prep QC Batch: MP97572



Report of Analysis

Page 1 of 1

Client Sample ID: OSMW-3S

Lab Sample ID:

Matrix:

Project:

JC33375-1

Date Sampled: 12/06/16 Date Received: 12/09/16

AQ - Ground Water

BMSMC, Building 5 Area, PR

Percent Solids: n/a

General Chemistry

Analyte	Result	RL	Units	DF	Analyzed	Ву	Method
Alkalinity, Total as CaCO3 Iron, Ferric ^a Iron, Ferrous ^b Nitrogen, Nitrate ^c Nitrogen, Nitrate + Nitrite Nitrogen, Nitrite ^d Sulfate Sulfide	214 3.0 < 0.20 < 0.11 < 0.10 < 0.010 22.1 < 2.0	5.0 0.30 0.20 0.11 0.10 0.010 10 2.0	mg/l mg/l mg/l mg/l mg/l mg/l mg/l	1 1 1 1 1 1 1	12/15/16 22:50 12/15/16 14:21 12/10/16 13:28 12/21/16 13:19 12/21/16 13:19 12/09/16 22:48 12/19/16 21:56 12/13/16 14:53	AB YR YZ YZ CB JN	SM2320 B-11 SM3500FE B-11 SM3500FE B-11 EPA353.2/SM4500NO2B EPA 353.2/LACHAT SM4500NO2 B-11 EPA 300/SW846 9056A SM4500S2- F-11

- (a) Calculated as: (Iron) (Iron, Ferrous)
- (b) Field analysis required. Received out of hold time and analyzed by request.
- (c) Calculated as: (Nitrogen, Nitrate + Nitrite) (Nitrogen, Nitrite) Nitrogen, Nitrite analysis done past holding time.
- (d) Sample received outside the holding time.



Page 1 of 1

Report of Analysis

Ву

RL

Prep Date

12/13/16

Client Sample ID: OSMW-4S Lab Sample ID: JC33375-2

File ID

P109787.D

Matrix:

AQ - Ground Water

DF

1

SGS Accutest LabLink@939893 10:02 27-Dec-2016

Method: Project:

SW846 8270D SW846 3510C

BMSMC, Building 5 Area, PR

Date Sampled: 12/06/16 Date Received: 12/09/16

Percent Solids: n/a

OP99167

Analytical Batch Prep Batch

EP4874

Run #1 Run #2

Final Volume Initial Volume Run #1 990 ml 1.0 ml

Run #2

RL CAS No. Compound Result MDL Units Q

Analyzed

12/14/16

100-52-7 Benzaldehyde ND 5.1 0.29 ug/l 123-91-1 1,4-Dioxane 31.5 1.0 0.66ug/l

CAS No. Surrogate Recoveries Run#1 Run#2 Limits

4165-60-0 Nitrobenzene-d5 32-128% 68% 321-60-8 2-Fluorobiphenyl 77% 35-119% 1718-51-0 Terphenyl-d14 92% 10-126%



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



SGS Accutest LabLink@939893 10:02 27-Dec-2016

Report of Analysis

Ву

SG

Page 1 of 1

Client Sample ID:	OSMW-4S
Lab Sample ID:	IC33375-2

File ID

3P57223.D

Matrix: Method: AQ - Ground Water

SW846 8270D BY SIM SW846 3510C

Analyzed

12/14/16

Date Sampled: Date Received:

12/06/16 12/09/16

Percent Solids:

Project:

BMSMC, Building 5 Area, PR

DF

1

Prep Date

12/13/16

Prep Batch OP99167A

Analytical Batch E3P2653

Run #1 Run #2

Initial Volume Final Volume Run #1 990 ml 1.0 ml

Compound

Run #2

CAS No.

Result RL MDL Units Q

56-55-3 Benzo(a)anthracene ND 0.051 0.023 ug/l 91-20-3 Naphthalene ND 0.10 0.030 ug/l

CAS No. Surrogate Recoveries Run#1 Run# 2 Limits

4165-60-0 Nitrobenzene-d5 71% 24-125% 321-60-8 2-Fluorobiphenyl 76% 19-127% 1718-51-0 Terphenyl-d14 67% 10-119%



E = Indicates value exceeds calibration range

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

SGS Accutest LabLink@939893 10:02 27-Dec-2016

Report of Analysis

Page 1 of 1

Client Sample ID: OSMW-4S Lab Sample ID:

JC33375-2

Matrix:

AQ - Ground Water

Method:

RSK-175

DF

5

Date Sampled:

Q

12/06/16 Date Received: 12/09/16

Percent Solids: n/a

Project:

BMSMC, Building 5 Area, PR

Prep Date

Prep Batch n/a

Analytical Batch GAA1095

Run #1 Run #2

CAS No.

74-82-8

Compound

Methane

AA56401.D

File ID

Result

246

Analyzed

12/16/16

RL

0.55

Ву

LM

MDL

0.18

n/a

Units

ug/l



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



Report of Analysis

Page 1 of 1

Client Sample ID: OSMW-4S

Lab Sample ID:

JC33375-2

Matrix: Method: Project:

AQ - Ground Water

SW846 8081B SW846 3510C

BMSMC, Building 5 Area, PR

Date Sampled:

12/06/16 Date Received: 12/09/16

Percent Solids: n/a

Analytical Batch DF Analyzed Prep Date Prep Batch By 1G130506.D 1 12/14/16 KD 12/13/16 OP99172 G1G4171

Run #1 Run #2

Initial Volume Run #1 940 ml

Final Volume 10.0 ml

Run #2

Compound

File ID

Result

RL

MDL

Units

0

60-57-1

CAS No.

Dieldrin

ND

0.011 0.0038ug/l

CAS No. Surrogate Recoveries

Run#1

90%

Run#2

Limits 26-132%

877-09-8 Tetrachloro-m-xylene 877-09-8 Tetrachloro-m-xylene 2051-24-3 Decachlorobiphenyl 2051-24-3 Decachlorobiphenyl

93% 77% 85%

26-132% 10-118% 10-118%



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

JC33375-2 AQ - Ground Water Date Sampled: 12/06/16 Date Received: 12/09/16

Matrix: Project:

BMSMC, Building 5 Area, PR

Percent Solids: n/a

Total Metals Analysis

Analyte	Result	RL	MDL	Units	DF	Prep	Analyzed By	Method	Prep Method
Iron Manganese	3020 454	100 15	12 0.39	ug/l ug/l			12/15/16 AB 12/15/16 AB	SW846 6010C ¹ SW846 6010C ¹	SW846 3010A ² SW846 3010A ²

(1) Instrument QC Batch: MA40966 (2) Prep QC Batch: MP97572



Report of Analysis

Client Sample ID: OSMW-4S Lab Sample ID: JC33375-2

AQ - Ground Water

Date Sampled: 12/06/16 Date Received: 12/09/16 Percent Solids: n/a

Matrix: Project:

BMSMC, Building 5 Area, PR

General Chemistry

Analyte	Result	RL	Units	DF	Analyzed	Ву	Method
Alkalinity, Total as CaCO3	335	5.0	mg/l	1	12/15/16 22:50	СВ	SM2320 B-11
Iron, Ferric ^a	2.9	0.30	mg/l	1	12/15/16 14:31	AB	SM3500FE B-11
Iron, Ferrous ^b	< 0.20	0.20	mg/l	1	12/10/16 13:35	YR	SM3500FE B-11
Nitrogen, Nitrate ^c	< 0.11	0.11	mg/l	1	12/21/16 13:20	YZ	EPA353.2/SM4500NO2B
Nitrogen, Nitrate + Nitrite	< 0.10	0.10	mg/l	1	12/21/16 13:20	YZ	EPA 353.2/LACHAT
Nitrogen, Nitrite d	< 0.010	0.010	mg/l	1	12/09/16 22:48	CB	SM4500NO2 B-11
Sulfate	< 10	10	mg/l	1	12/19/16 22:20	JN	EPA 300/SW846 9056A
Sulfide	< 2.0	2.0	mg/l	1	12/13/16 14:53	JA	SM4500S2- F-11

- (a) Calculated as: (Iron) (Iron, Ferrous)
- (b) Field analysis required. Received out of hold time and analyzed by request.
- (c) Calculated as: (Nitrogen, Nitrate + Nitrite) (Nitrogen, Nitrite) Nitrogen, Nitrite analysis done past holding time.
- (d) Sample received outside the holding time.



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JC33375: Chain of Custody Page 1 of 3

EXECUTIVE NARRATIVE

SDG No:

JC33375

Laboratory:

Accutest, New Jersey

Analysis:

SW846-8270D

Number of Samples:

2

Location:

BMSMC, Building 5 Area

Humacao, PR

SUMMARY: Two (2) samples were analyzed for selected SVOCs following method SW846-8270D and Selected PAHs and 1,4-Dioxane were also analyzed by SW846-8270D using the selective ion monitoring (SIM) technique. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: EPA Hazardous Waste Support Section, SOP HW-35A, July 2015 –Revision 0. Semivolatile Data Validation. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues:

None

Major:

None

Minor:

None

Critical findings:

None

Major findings:

None

Minor findings:

1. Initial and continuing calibration verifications meet the method and guidance document required performance criteria except in the cases described in the Data Review Worksheet. Results for Benzo(a)anthracene were qualified as estimated (J or UJ) in affected samples.

No closing calibration verification included in data package. No action taken, professional

judgment.

QC samples were not validated.

COMMENTS:

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

January 14, 2017

SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: JC33375-1

Sample location: BMSMC Building 5 Area

Sampling date: 6-Dec-16

Matrix: Groundwater

METHOD: 8270D

Analyte Name Result Units Dilution Factor Lab Flag Validation Reportable Benzaldehyde 5.0 ug/l 1 - U Yes

METHOD: 8270D (SIM)

Lab Flag Validation Reportable **Analyte Name** Result Units Dilution Factor Benzo(a)anthracene 1 Yes v 0.050 UJ ug/l 1 U Naphthalene 0.10 ug/l Yes 1 1,4-Dioxane 1.73 Yes ug/l

Sample ID: JC33375-2

Sample location: BMSMC Building 5 Area

Sampling date: 6-Dec-16

Matrix: Groundwater

METHOD: 8270D

Analyte Name Result Units Dilution Factor Lab Flag Validation Reportable
Benzaldehyde 5.1 ug/l 1 - U Yes
1,4-Dioxane 31.5 ug/l 1 - Yes

METHOD: 8270D (SIM)

Units Dilution Factor Lab Flag Validation Reportable **Analyte Name** Result Yes 🗸 Benzo(a)anthracene 0.051 1 UJ ug/l Naphthalene 0.10 ug/l 1 U Yes

	Date:December_6
REVIEW OF SEMIVOLATILE C	
The following guidelines for evaluating volatile orgalidation actions. This document will assist the remake more informed decision and in better serving results were assessed according to USEPA data following order of precedence: EPA Hazardous W 2015 – Revision 0. Semivolatile Data Validation. The QC on the data review worksheets are from the prima noted.	eviewer in using professional judgment to the needs of the data users. The sample a validation guidance documents in the laste Support Section, SOP HW-35A, July C criteria and data validation actions listed
The hardcopied (laboratory name) _Accutest	
Lab. Project/SDG No.:JC33375	
X Holding TimesX GC/MS TuningX Internal Standard Performance	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
_Overall Comments:_Selected_SVOCs_from_the_TCL_s _8270D;_Selected_PAHs _and_1,4-Dioxane_analyzed_b; _Field_and_Equipment_Blanks_validated_in_another_job	y_method_SW846-8270D_(SIM)
Definition of Qualifiers:	
J- Estimated results U- Compound not detected R- Rejected data UJ- Estimated nondetect Reviewer: Alan Manual Date: January 14, 2017	

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
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38300		
		<u></u>
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		_
[V		
	289	

All criteria were met _	_X_	
Criteria were not met		
and/or see below		

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED/ANALYZED	рН	ACTION						
All samples extracted and analyzed within method recommended holding.										

Cooler temperature	(Criteria: 4 + 2 °C)): 5.2°C	

<u>Actions</u>

Results will be qualified based on the criteria of the following Table:

Table 1. Holding Time Actions for Semivolatile Analyses

			Ac	tion
Matrix	Preserved	Criteria	Detected Associated Compounds	Non-Detected Associated Compounds
No		≤7 days (for extraction) ≤40 days (for analysis)	Use professi	onal judgment
	No	> 7 days (for extraction) > 40 days (for analysis)	J	Use professional judgment
Aqueous	Yes	≤ 7 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No	Grossly Exceeded	J	UJ or R
	No	≤ 14 days (for extraction) ≤ 40 days (for analysis)	Use profession	onal judgment
Non-Aqueous	No	> 14 days (for extraction) > 40 days (for analysis)	J	Use professional judgment
	Yes	≤ 14 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No	Grossly Exceeded	J	UJ or R

All criteria were met _	_X	
Criteria were not met see below		_

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

_X__ The DFTPP performance results were reviewed and found to be within the specified criteria.

_X__ DFTPP tuning was performed for every 12 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

Notes: These requirements do not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.

All mass spectrometer conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortion are unacceptable

Notes: No data should be qualified based of DFTPP failure.

The requirement to analyze the instrument performance check solution is optional when analysis of PAHs/pentachlorophenol is to be performed by the SIM technique.

List	the	samples	affected:

Actions:

- 1. If sample are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check, qualify all data in those samples as unusable (R).
- 2. If ion abundance criteria are not met, use professional judgment to determine to what extent the data may be utilized.
- 3. State in the Data Review Narrative, decisions to use analytical data associated with DFTPP instrument performance checks not meeting the contract requirements.
- 4. Use professional judgment to determine if associated data should be qualified based on the spectrum of the mass calibration compounds.

All criteria were met	_X	
Criteria were not met		
and/or see below		

INITIAL CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Instrume	nt ID nur	mbers:_	10/18/16_(SIM) GCMS3P Aqueous/low			
Instrume	nt ID nur	nbers:_	_12/08/16_(SCAN) GCMS3E Aqueous/low_		GCMS6P_	(SCAN)
Instrume	nt ID nur	nbers:_	_11/28-29/16_(SCAN GCMSP Aqueous/low_			
DATE	LAB	FILE	CRITERIA OUT	COMPOUND		SAMPLES

		ID#	RFs, %RSD, %D, r		AFFECTED
ĺ					
	Initial a	and initial calib	ration verification mee	ts the method and guidance va	lidation document
			perforn	nance criteria.	

Note:

Actions:

Qualify the initial calibration analytes listed in Table 2 using the following criteria:

Table 3. Initial Calibration Actions for Semivolatile Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
Initial Calibration not performed at specified frequency and sequence	Use professional judgment	Use professional judgment R	
Initial Calibration not performed at the specified concentrations	J	υJ	
RRF < Minimum RRF in Table 2 for target analyte	Use professional judgment J+ or R	R	
RRF ≥ Minimum RRF in Table 2 for target analyte	No qualification	No qualification	
%RSD > Maximum %RSD in Table 2 for target analyte	1	Use professional judgment	
%RSD ≤ Maximum %RSD in Table 2 for target analyte	No qualification	No qualification	

Initial Calibration

Table 2. RRF, %RSD, and %D Acceptance Criteria in Initial Calibration and CCV for Semivolatile Analysis

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D ¹	Opening Maximum %D ¹
1,4-Dioxane	0.010	40.0	± 40.0	± 50.0
Benzaldehyde	0.100	40.0	± 40.0	± 50.0
Phenol	0.080	20.0	± 20.0	± 25.0
Bis(2-chloroethyl)ether	0.100	20.0	± 20.0	± 25.0
2-Chlorophenol	0.200	20.0	± 20.0	± 25.0
2-Methylphenol	0.010	20.0	± 20.0	±25.0
3-Methylphenol	0.010	20.0	± 20.0	± 25.0
2,2'-Oxybis-(1-chloropropane)	0.010	20.0	± 25.0	± 50.0
Acetophenone	0.060	20.0	± 20.0	± 25.0
4-Methylphenol	0.010	20.0	±20.0	±25.0
N-Nitroso-di-n-propylamine	0.080	20.0	±25.0	± 25.0
Hexachloroethane	0.100	20.0	±20.0	± 25.0
Nitrobenzene	0.090	20.0	±20.0	±25.0
Isophorone	0.100	20.0	± 20.0	± 25.0
2-Nitrophenol	0.060	20.0	±20.0	± 25.0
2,4-Dimethylphenol	0.050	20.0	± 25.0	± 50.0
Bis(2-chloroethoxy)methane	0.080	20.0	±20.0	± 25.0
2,4-Dichlorophenol	0.060	20.0	±20.0	±25.0
Naphthalene	0.200	20.0	± 20.0	± 25.0
4-Chloroaniline	0.010	40.0	± 40.0	± 50.0
Hexachlorobutadiene	0.040	20.0	± 20.0	±25.0
Caprolactam	0.010	40.0	±30.0	± 50.0
4-Chloro-3-methylphenol	0.040	20.0	± 20.0	± 25.0
2-Methylnaphthalene	0.100	20.0	± 20.0	±25.0
l lexachlorocyclopentadiene	0.010	40.0	± 40.0	± 50.0
2,4,6-Trichlorophenol	0.090	20.0	±20.0	± 25.0
2,4,5-Trichlorophenol	0.100	20.0	± 20.0	± 25.0
1,1'-Biphenyl	0.200	20.0	± 20.0	±25.0

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D ¹	Opening Maximum %D ¹
2-Chloronaphthalene	0.300	20.0	±20.0	±25.0
2-Nitroaniline	0.060	20.0	± 25.0	± 25.0
Dimethylphthalate	0.300	20.0	± 25.0	± 25.0
2,6-Dinitrotoluene	0.080	20.0	± 20.0	± 25.0
Acenaphthylene	0.400	20.0	±20.0	± 25.0
3-Nitroaniline	0.010	20.0	±25.0	± 50.0
Acenaphthene	0.200	20.0	±20.0	± 25.0
2,4-Dinitrophenol	0.010	40.0	± 50.0	± 50.0
4-Nitrophenol	0.010	40.0	± 40.0	± 50.0
Dibenzofuran	0.300	20.0	± 20.0	± 25.0
2,4-Dinitrotoluene	0.070	20.0	± 20.0	± 25.0
Diethylphthalate	0.300	20.0	±20.0	± 25.0
1,2,4,5-Tetrachlorobenzene	0.100	20.0	±20.0	± 25.0
4-Chlorophenyl-phenylether	0.100	20.0	± 20.0	± 25.0
Fluorene	0.200	20.0	± 20.0	± 25.0
4-Nitroaniline	0.010	40.0	±40.0	± 50.0
4,6-Dinitro-2-methylphenol	0.010	40.0	±30.0	± 50.0
4-Bromophenyl-phenyl ether	0.070	20.0	± 20.0	± 25.0
N-Nitrosodiphenylamine	0.100	20.0	± 20.0	± 25.0
Hexachlorobenzene	0.050	20.0	± 20.0	± 25.0
Atrazine	0.010	40.0	±25.0	± 50.0
Pentachlorophenol	0.010	40.0	± 40.0	± 50.0
Phenanthrene	0.200	20.0	± 20.0	± 25.0
Anthracene	0.200	20.0	± 20.0	± 25.0
Carbazole	0.050	20.0	±20.0	± 25.0
Di-n-butylphthalate	0.500	20.0	± 20.0	± 25.0
Fluoranthene	0.100	20.0	±20.0	± 25.0
Pyrene	0.400	20.0	±25.0	± 50.0
Butylbenzylphthalate	0.100	20.0	±25.0	± 50.0

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D ¹	Opening Maximum %D¹
3,3'-Dichlorobenzidine	0.010	40.0	± 40.0	± 50.0
Benzo(a)anthracene	0.300	20.0	±20.0	± 25.0
Chrysene	0.200	20.0	± 20.0	± 50.0
Bis(2-ethylhexyl) phthalate	0.200	20.0	±25.0	± 50.0
Di-n-octylphthalate	0.010	40.0	± 40.0	± 50.0
Benzo(b)fluoranthene	0.010	20.0	±25.0	± 50.0
Benzo(k)fluoranthene	0.010	20.0	±25.0	± 50.0
Benzo(a)pyrene	0.010	20.0	±20.0	± 50.0
Indeno(1,2,3-cd)pyrene	0.010	20.0	±25.0	± 50.0
Dibenzo(a,h)anthracene	0.010	20.0	±25.0	± 50.0
Benzo(g,h,i)perylene	0.010	20.0	±30.0	± 50.0
2,3,4,6-Tetrachlorophenol	0.040	20.0	± 20.0	± 50.0
Naphthalene	0.600	20.0	± 25.0	± 25.0
2-Methylnaphthalene	0.300	20.0	± 20.0	± 25.0
Acenaphthylene	0.900	20.0	±20.0	±25.0
Acenaphthene	0.500	20.0	±20.0	± 25.0
Fluorene	0.700	20.0	±25.0	± 50.0
Phenanthrene	0.300	20.0	±25.0	± 50.0
Anthracene	0.400	20.0	±25.0	± 50.0
Fluoranthene	0.400	20.0	±25.0	± 50.0
Pyrene	0.500	20.0	± 30.0	± 50.0
Benzo(a)anthracene	0.400	20.0	±25.0	± 50.0
Chyrsene	0.400	20.0	±25.0	± 50.0
Benzo(b)fluoranthene	0.100	20.0	± 30.0	± 50.0
Benzo(k)fluoranthene	0.100	20.0	±30.0	± 50.0
Benzo(a)pyrene	0.100	20.0	±25.0	± 50.0
Indeno(1,2,3-cd)pyrene	0.100	20.0	± 40.0	± 50.0
Dibenzo(a,h)anthracene	0.010	25.0	± 40.0	±50.0
Benzo(g,h,i)perylene	0.020	25.0	± 40.0	± 50.0

Pentachlorophenol	0.010	40.0	± 50.0	± 50.0	
Deuterated Monitoring Compounds					

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D ¹	Closing Maximum %D
1,4-Dioxane-d ₈	0.010	20.0	± 25.0	± 50.0
Phenol-d ₅	0.010	20.0	± 25.0	± 25.0
Bis-(2-chloroethyl)ether-d ₈	0.100	20.0	± 20.0	±25.0
2-Chlorophenol-d4	0.200	20.0	± 20.0	±25.0
4-Methylphenol-d ₈	0.010	20.0	± 20.0	±25.0
4-Chloroaniline-d ₄	0.010	40.0	± 40.0	±50.0
Nitrobenzene-d ₅	0.050	20.0	± 20.0	±25.0
2-Nitrophenol-d ₄	0.050	20.0	±20.0	± 25.0
2,4-Dichlorophenol-d3	0.060	20.0	± 20.0	±25.0
Dimethylphthalate-d ₆	0.300	20.0	± 20.0	±25.0
Acenaphthylene-d ₈	0.400	20.0	± 20.0	±25.0
4-Nitrophenol-d ₄	0.010	40.0	± 40.0	±50.0
Fluorene-d ₁₀	0.100	20.0	± 20.0	±25.0
4,6-Dinitro-2-methylphenol-d2	0.010	40.0	± 30.0	± 50.0
Anthracene-d ₁₀	0.300	20.0	± 20.0	±25.0
Pyrene-d ₁₀	0.300	20.0	± 25.0	± 50.0
Benzo(a)pyrene-d ₁₂	0.010	20.0	±20.0	± 50.0
Fluoranthene-d ₁₀ (SIM)	0.400	20.0	± 25.0	± 50.0
2-Methylnaphthalene-d ₁₀ (SIM)	0.300	20.0	± 20.0	± 25.0

¹ If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

Note: If analysis by SIM technique is requested for PAH/pentachlorophenols, calibration standards analyzed at 0.10, 0.20, 0.40, 0.80, and 1.0 ng/uL for each target compound of interest and the associated DMCs. Pentachlorophenol will require only a four point initial calibration at 0.20, 0.40, 0.80, and 1.0 ng/uL.

All criteria were met	
Criteria were not met	
and/or see belowX	

CONTINUING CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	10/18/16_(SIM)	
	ation (ICV):10/19/16	
Date of continuing calibration v	erification (CCV):_12/14/16;_12/17/16	5
Date of closing CCV:	•	
Instrument ID numbers:	GCMS3P	
Matrix/Level:	Aqueous/low	
Date of initial calibration:	11/28-29/16_(Scan)	11/18/16_(Scan)
	ation (ICV):_11/29/306	
	erification (CCV):_12/14/16	
Date of closing CCV:		-
Instrument ID numbers:	- GCMSP	GCMS6P
Matrix/Level:	Aqueous/low	Aqueous/low
Date of initial calibration:	12/08/16_(Scan)	_
Date of initial calibration verification	ation (ICV):_12/08-09/16	
Date of continuing calibration v	erification (CCV):_12/15/16	_
Date of closing CCV:	<u>-</u>	
Instrument ID numbers:	GCSM3E	
Matrix/Level:	Aqueous/low	<u></u>

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, %D , r	COMPOUND	SAMPLES AFFECTED
GCMS3P	IUT	111 3, 78110D, <u>78D,</u> 1		AITECIED
12/14/16	cc2579-0.5	-42.3	Benzo(a)anthracene	JC33375-1; -2

Note: Initial and continuing calibration verifications meet the method and guidance document required performance criteria except for the cases described in this document. Results qualified as estimated (J or UJ) in affected samples.

performance criteria but within the guidance document performance criteria. No action taken.

No action taken for QC samples.

No closing calibration verification included in data package. No action taken, professional judgment.

Actions:

Notes: Verify that the CCV is run at the required frequency (an opening and closing CCV must be run within 12-hour period).

All DMCs must meet the RRF values given in Table 2. No qualification of the data is necessary on DMCs RRF and %RSD/%D alone. Use professional judgment to evaluate DMCs and %RSD/%D data in conjunction with DMCs recoveries to determine the need for qualification of the data.

Qualify the initial calibration analytes listed in Table 2 using the following criteria in the CCVs:

Table 4. CCV Actions for Semivolatile Analysis

Criteria for Opening CCV	Criteria for Closing CCV	Action		
Criteria for Opening CCV	Criteria for Closing CCV	Detect	Non-detect	
CCV not performed at required frequency and sequence	CCV not performed at required frequency	Use professional judgment R	Use professional judgment R	
CCV not performed at specified concentration	CCV not performed at specified concentration	Use professional judgment	Use professional judgment	
RRF < Minimum RRF in Table 2 for target analyte	RRF Minimum RRF in Table 2 for target analyte	Use professional judgment J or R	R	
RRF ≥ Minimum RRF in Table 2 for target analyte	RRF ≥ Minimum RRF in Table 2 for target analyte	No qualification	No qualification	
%D outside the Opening Maximum %D limits in Table 2 for target analyte	%D outside the Closing Maximum %D limits in Table 2 for target analyte	J	บา	
%D within the inclusive Opening Maximum %D limits in Table 2 for target analyte	%D within the inclusive Closing Maximum %D limits in Table 2 for target analyte	No qualification	No qualification	

All criteria were met _	_X	_
Criteria were not met		
and/or see below		

BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Notes: The concentration of non-target compounds in all blanks must be less than or equal to 10 ug/L.

The concentration of target compounds in all blanks must be less than its CRQL listed in the method.

Samples taken from a drinking water tap do not have and associated field blank.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No_target_ana	2020			
Note:				
Field/Equipmer	nt/Trip blank			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
				zed_with_this_data_package
	3.8.05			
·				
Note:				

All criteria were met __X_ Criteria were not met

andles		holow	
and/or	566	Delow	_

BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Qualify samples based on the criteria summarized in Table 5:

Table 5. Blank and TCLP/SPLP LEB Actions for Semivolatile Analysis

Blank Type	Blank Result	Sample Result	Action
	Detect	Non-detect	No qualification
	< CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL	Use professional judgment
		< CRQL	Report at CRQL and qualify as non-detect (U)
Method,	≥CRQL	≥ CRQL but < Blank Result	Report at sample results and qualify as non-detect (U) or as unusable (R)
TCLP/SPLP LEB, Field		≥ CRQL and ≥ Blank Result	Use professional judgment
· 	Grossly high	Detect	Report at sample results and qualify as unusable (R)
	TIC > 5.0 ug/L (water) or 0.0050 mg/L (TCLP leachate) or TIC > 170 ug/Kg (soil)	Detect	Use professional judgment

List samples qualified

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
			' 		

All criteria were met __X___ Criteria were not met

and/or	see	he	οw	
	366	20	UW	_

SURROGATE SPIKE RECOVERIES - DEUTERATED MONITORING COMPOUNDS (DMCs)

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries – deuterated monitoring compounds. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

Notes: Recoveries for DMCs in samples and blanks must be within the limits specified in Table 6.

The recovery limits for any of the compounds listed in Table 6 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

If a DMC is not added in the samples and blanks or the concentrations of DMCs in the samples and blank not the specified, use professional judgment in qualifying the data.

Cuitania	Action		
Criteria	Detect	Non-detect	
%R < 10% (excluding DMCs with 10% as a lower acceptance limit)	J-	R	
10% ≤ %R (excluding DMCs with 10% as a lower acceptance limit) < Lower Acceptance Limit	J-	UJ	
Lower Acceptance limit \leq %R \leq Upper Acceptance Limit	No qualification	No qualification	
%R > Upper Acceptance Limit	JĤ	No qualification	

Table 7. DMC Actions for Semivolatile Analysis

List the percent recoveries (%Rs) which do not meet the criteria for DMCs (surrogate) recovery.

Matrix:Groundwater_		
SAMPLE ID	SURROGATE COMPOUND	ACTION
	ed_criteria_in_all_samples_analyzedNonde_and_were_within_laboratory_recovery_limits	_

- (a) Outside control limits due to matrix interference.
- (b) Outside in house control limits biased low. The results confirmed by re-extraction outside the holding time.

Note:

Table 8. Semivolatile DMCs and the Associated Target Analytes

1.4.5: 1.45140.43	DI LI (DIEC 2)		
1,4-Dioxane-d ₈ (DMC-1)	Phenol-d ₅ (DMC-2)	Bis(2-Chloroethyl) ether-d ₈ (DMC-3)	
1,4-Dioxane	Benzaldehyde	Bis(2-chloroethyl)ether	
117 6217/16116	Phenol	2,2'-Oxybis(1-chloropropane)	
	7.1101101	Bis(2-chloroethoxy)methane	
2-Chlorophenol-d4(DMC-4)	4-Methylphenol-da (DMC-5)	4-Chloroaniline-d ₄ (DMC-6)	
2-Chlorophenol	2-Methylphenol	4-Chloroaniline	
2-Chlorophenot	3-Methylphenol	Hexachlorocyclopentadiene	
	4-Methylphenol	Dichlorobenzidine	
	2,4-Dimethylphenol	Diemorobenziame	
		A D 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
Nitrobenzene-d ₅ (DMC-7)	2-Nitrophenol-d ₄ (DMC-8)	2,4-Dichlorophenol-d ₃ (DMC-9)	
Acetophenone	Isophorone	2,4-Dichlorophenol	
N-Nitroso-di-n-propylamine	2-Nitrophenol	Hexachlorobutadiene	
Hexachloroethane		Hexachlorocyclopentadiene	
Nitrobenzene		4-Chloro-3-methylphenol	
2,6-Dinitrotoluene		2,4,6-Trichlorophenol	
2,4-Dinitrotoluene		2,4,5-Trichlorophenol	
N-Nitrosodiphenylamine		1,2,4,5-Tetrachlorobenzene	
• •		*Pentachlorophenol	
		2,3,4,6-Tetrachlorophenol	
Dimethylphthalate-d ₆ (DMC-10)	Acenaphthylene-da (DMC-11)	4-Nitrophenol-d ₄ (DMC-12)	
Caprolactam	*Naphthalene	2-Nitroaniline	
1,1'-Biphenyl	*2-Methylnaphthalene	3-Nitroaniline	
Dimethylphthalate	2-Chloronaphthalene	2,4-Dinitrophenol	
Diethylphthalate	*Acenaphthylene	4-Nitrophenol	
Di-n-butylphthalate	*Acenaphthene	4-Nitroaniline	
Butylbenzylphthalate			
Bis(2-ethylhexyl) phthalate			
Di-n-octylphthalate			

Fluorenc-d ₁₀ (DMC-13)	4,6-Dinitro-2-methylphenol-d ₂ (DMC-14)	Anthracene-d ₁₀ (DMC-15)
Dibenzofuran *Fluorene	4,6-Dinitro-2-methylphenol	Hexachlorobenzene Atrazine
4-Chlorophenyl-phenylether		*Phenanthrene
4-Bromophenyl-phenylether		*Anthracene
Carbazole		
Pyrene-d ₁₀ (DMC-16)	Benzo(a)pyrene-d ₁₂ (DMC-17)	
*Fluoranthene	3,3'-Dichlorobenzidine	
*Pyrene	*Benzo(b)fluoranthene	
*Benzo(a)anthracene	*Benzo(k)fluoranthene	
*Chrysene	*Benzo(a)pyrene	
	*Indeno(1,2,3-cd)pyrene	
	*Dibenzo(a,h)anthracene	
	*Benzo(g,h,i)perylene	

^{*}Included in optional Target Analyte List (TAL) of PAHs and PCP only.

Table 9. Semivolatile SIM DMCs and the Associated Target Analytes

Fluoranthene-d10 (DMC-1)	2-Methylnaphthalene-d10 (DMC-2)
Fluoranthene	Naphthalene
Pyrene	2-Methylnaphthalene
Benzo(a)anthracene	Acenaphthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

All criteria were met	_X	
Criteria were not met		
and/or see below	- 5	

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

NOTES:

Data for MS and MSDs will not be present unless requested by the Region. Notify the Contract Laboratory COR if a field or trip blank was used for the MS and MSD.

For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:_	JC33175-1	Matrix/Level:	Groundwater
Sample ID:_	JC33175-1_(SIM)	Matrix/Level:	Groundwater

Note: MS/MSD % recoveries and RPD within laboratory control limits.

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J). If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met __X__ Criteria were not met and/or see below____

INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

DATE SAMPLE ID IS OUT IS AREA ACCEPTABLE ACTION RANGE

Internal area meets the required criteria for batch samples corresponding to this data package.

Action:

- 1. If an internal standard area count for a sample or blank is greater than 200% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 10 below):
 - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
 - b. Do not qualify non-detected associated compounds.
- 2. If an internal standard area count for a sample or blank is less than 20.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
 - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
 - b. Qualify non-detected associated compounds as unusable (R).
- 3. If an internal standard area count for a sample or blank is greater than or equal to 50.0%, and less than or equal to 200% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
- 4. If an internal standard RT varies by more than 10.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
- 5. If an internal standard RT varies by less than or equal to 10.0 seconds, no qualification of the data is necessary.

Note: Inform the Contract Laboratory Program Project Officer (CLP PO) if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

State in the Data Review Narrative if the required internal standard compounds are not added to a sample or blank or if the required internal standard compound is not analyzed at the specified concentration.

Actions:

Table 10. Internal Standard Actions for Semivolatile Analysis

Criteria	Action	
Спена	Detect	Non-detect
Area response < 20% of the opening CCV or mid-point standard CS3 from ICAL	j+	R
20% ≤ Area response < 50% of the opening CCV or mid-point standard CS3 from ICAL	J+	UJ
50% ≤ Area response ≤ 200% of the opening CCV or mid-point standard CS3 from ICAL	No qualification	No qualification
Area response > 200% of the opening CCV or mid-point standard CS3 from ICAL	J-	No qualification
RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL > 10.0 seconds	R	R
RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL < 10.0 seconds	No qualification	No qualification

		All criteria were metX Criteria were not met and/or see below
TARGET CO	MPOUND IDENTIFICATION	
Criteria:		
		ounds within ±0.06 RRT units of the standard CV) or mid-point standard from the initia Yes? or No?
List compour	nds not meeting the criteria described above:	
Sample ID	Compounds	Actions
spectrum fro	om the associated calibration standard (oper must match according to the following criteria: All ions present in the standard mass sper must be present in the sample spectrum. The relative intensities of these ions must sample spectra (e.g., for an ion with an the corresponding sample ion abundance lons present at greater than 10% in the samples.	ectrum at a relative intensity greater than 10% agree within ±20% between the standard and abundance of 50% in the standard spectrum.
List compour	nds not meeting the criteria described above:	
Sample ID	Compounds	Actions
_Identified_c	ompounds_meet_the_required_criteria	

Action:

- 1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
- 2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
- Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory COR action, the necessity for numerous or significant changes.

TENTATIVELY IDENTIFIED COMPOUNDS (TICS)

NOTE: Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

			 $\overline{}$
	is	r	 Cs
4 1			

Sample ID	Compound	Sample ID	Compound
	=======================================	=======================================	

Action:

- 1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations. TICs labeled "unknown" are qualified as estimated (J).
- 2. General actions related to the review of TIC results are as follows:
 - a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).
 - b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
- 3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
- 4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

- 5. Target compounds from other fractions and suspected laboratory contaminants should be marked as "non-reportable".
- 6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
- 7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
- 8. Note, for Contract Laboratory COR action, failure to properly evaluate and report TICs

All criteria were met _	_X	
Criteria were not met		
and/or see below		

SAMPLE QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

Action:

- 1. When a sample is analyzed at more than one dilution, the lower CRQL are used unless a QC exceedance dictates the use of higher CRQLs from the diluted sample. Samples reported with an "E" qualifier should be reported from the diluted sample.
- 2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
- 3. For non-aqueous samples, if the solids is less than 10.0%, use professional judgment for both detects and non-detects. If the percent solid for a soil sample is greater than or equal to 10.0% and less than 30.0%, use professional judgment to qualify detects and non-detects. If the percent solid for a soil sample is greater than or equal to 30.0%, detects and non-detects should not be qualified (see Table 11).
- 4. Note, for Contract Laboratory COR action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.
- 5. Results between MDL and CRQL should be qualified as estimated "J".
- 6. Results < MDL should be reported at the CRQL and qualified "U". MDLs themselves should not be reported.

Table 11. Percent Solids Actions for Semivolatile Analysis for Non-Aqueous Samples

Criteria	Ac	Action		
Criteria	Detects	Non-detects		
%Solids < 10.0%	Use professional judgment	Use professional judgment		
10.0% ≤ %Solids ≤ 30.0%	Use professional judgment	Use professional judgment		
%Solids > 30.0%	No qualification	No qualification		

SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
	ile -	
	- 4	
	1	
	-	
(GC)		

target analytes above 5 SQL.

				Criter	teria were met ia were not met r see belowN/A	
FIELD DUPLICATE	PRECIS	ION				
Sample IDs		<u> </u>	_	Mat	rix:	
Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples. The project QAPP should be reviewed for project-specific information. Suggested criteria: if large RPD (> 50 %) is observed, confirm identification of the samples and note differences. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.						
COMPOUND	SQL ug/L	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION	
No field/laboratory of	luplicate	analyzed as part	of this data package.	MS/MSI	D % recoveries	RPD

used to assess precision. RPD within the required guidance document criteria < 50 % for detected

All criteria were metX	
Criteria were not met	
and/or see below	

OTHER ISSUES

A.	System Perfo	ormance	
List s	amples qualified	based on the degradation of system	performance during simple analysis:
Sam	ple ID	Comments	Actions
durin	professional judo g sample analy	gment to qualify the data if it is determ	nined that system performance has degraded y Program COR any action as a result of
В.	·	ssment of Data	
List s	samples qualified	I based on other issues:	
Sam	ple ID	Comments	Actions
			_dataResults_are_valid_and_can_be_used n_below
 Note	*		
Actio	n:		

- 1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
- 2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Inform the Contract Laboratory COR the action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

- 3. Sometimes, due to dilutions, re-analysis or SIM/Scan runs are being performed, there will be multiple results for a single analyte from a single sample. The following criteria and professional judgment are used to determine which result should be reported:
 - The analysis with the lower CRQL
 - The analysis with the better QC results
 - The analysis with the higher results

MEMORANDUM

TO: Mr. Haley Royer

Anderson, Mulholland and Associates

DATE: January 14, 2017

FROM: R. Infante

FILE: JC33375

RE:

Data Validation

SDG: JC33375

SUMMARY

Full validation was performed on the data for two groundwater samples analyzed for dissolved methane by method RSK-175. The samples were collected at the Bristol Myer Squib-Building 5 Area, Humacao, PR site on December 06, 2016 and submitted to Accutest Laboratories of Dayton, New Jersey that analyzed and reported the results under delivery groups (SDG) JC33375. The sample results were assessed according to USEPA general data validation guidance documents.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
OSMW-3S	JC33375-1	12/06/16	Groundwater	Methane
OSMW-4S	JC33375-2	12/06/16	Groundwater	Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations
- o Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria
- Surrogate spike recovery
- o Internal standard performance and retention times
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody.

Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

Initial and Continuing Calibrations

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

No trip/field/equipment blank analyzed with this data package.

Laboratory/Field Duplicate Results

Field duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of +25% for analytes $5\times SQL$.

LCS/LCSD Results

LCS (blank spike) was analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were not performed.

Calculations were spot checked.

Summary

Samples JC33375-1 and JC33375-2 were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

Rafael Infante

Chemist License 1888

SAMPLE METHANE DATA SAMPLE SUMMARY

Sample ID: JC33375-1

Sample location: BMSMC Building 5 Area

Sampling date: 6-Dec-16

Matrix: Groundwater

METHOD: RSK -175

Analyte Name Result Units Dilution Factor Lab Flag Validation Reportable

Methane 3.2 ug/l 1 - - Yes

Sample ID: JC33375-2

Sample location: BMSMC Building 5 Area

Sampling date: 6-Dec-16

Matrix: Groundwater

METHOD: RSK-175

Analyte Name Result Units Dilution Factor Lab Flag Validation Reportable

Methane 246 ug/l 1 - Yes

EXECUTIVE NARRATIVE

SDG No:

JC33375

Laboratory:

Accutest, New Jersey

Analysis:

SW846-8081B

Number of Samples:

Location:

BMSMC, Building 5 Area

Humacao, PR

SUMMARY:

Two (2) samples were analyzed for selected pesticides (Dieldrin) following method SW846-8081B. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence Hazardous Waste Support Section SOP No. HW-36A, Revision O, June, 2015. SOM02.2. Pesticide Data Validation. The QC criteria and data validation actions listed on the data review worksheets are

from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues:

None

Major:

None

Minor:

None

Critical findings:

None

Major findings:

None

Minor findings:

None

COMMENTS:

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

January 14, 2017

PESTICIDE DATA SAMPLE SUMMARY

Sample ID: JC33375-1

Sample location: BMSMC Building 5 Area

Sampling date: 6-Dec-16

Matrix: Groundwater

METHOD: 8081B

Analyte Name Result Units Dilution Factor Lab Flag Validation Reportable

Dieldrin 0.010 ug/l 1 - U Yes

Sample ID: JC33375-2

Sample location: BMSMC Building 5 Area

Sampling date: 6-Dec-16

Matrix: Groundwater

METHOD: 8081B

Analyte Name Result Units Dilution Factor Lab Flag Validation Reportable

Dieldrin 0.011 ug/l 1 - U Yes

	Project/Case Number:JC33375 Sampling Date:12/06/2016 Shipping Date:12/08/2016 EPA Region No.:2
REVIEW OF PESTICIDE	ORGANIC PACKAGE
The following guidelines for evaluating volar required validation actions. This document wijudgment to make more informed decision as users. The sample results were assessed according to the following order of precedence of the following order ord	Ill assist the reviewer in using professional and in better serving the needs of the data cording to USEPA data validation guidance to Hazardous Waste Support Section SOP No. esticide Data Validation. The QC criteria and
The hardcopied (laboratory name) _Accutest reviewed and the quality control and performance data s	data package received has been ummarized. The data review for VOCs included:
Lab. Project/SDG No.:JC33375	
N/A GC/MS TuningX Internal Standard PerformanceX BlanksX Surrogate RecoveriesX Matrix Spike/Matrix Spike Duplicate	X CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Overall Comments:TCL_pesticides_list_(Dieldrin) _Field_and_Equipment_blanks_validated_in_anothe	
	ompound not detected stimated nondetect
Date: January 14, 2017	

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
1		
-		
4		
- 1		
	j.	·
	7.07	
		- 20 - 2000
		-
		-
		A.
	Service Servic	- W

All criteria were met _	_X_	
Criteria were not met		
and/or see below	_	

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE	DATE	ACTION
	SAMPLED	EXTRACTED/ANALYZED	
Samples properly pr	eserved. All sample	es extracted and analyzed wit	thin the required criteria.

Note:

<u>Criteria</u>

Aqueous samples - seven (7) days from sample collection for extraction; 40 days from sample collection for analysis.

Non-aqueous samples – fourteen (14) days from sample collection for extraction; 40 days from sample collection for analysis.

Cooler temperature (Criteria: 4 ± 2 °C): 5.2°C - OK

<u>Actions</u>

Qualify aqueous sample results using preservation and technical holding time information as follows:

- a. If there is no evidence that the samples were properly preserved (T = 4° C \pm 2° C), and the samples were extracted or analyzed within the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ).
- b. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}C \pm 2^{\circ}C$), and the samples were extracted or analyzed outside the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ).
- c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times, no qualification of the data is necessary.
- d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

- e. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
- f. If technical holding times are grossly exceeded, use professional judgment to qualify the data.

Qualify non-aqueous sample results using preservation and technical holding time information as follows:

- a. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}C \pm 2^{\circ}C$), and the samples were extracted or analyzed within the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
- b. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}C \pm 2^{\circ}C$), and the samples were extracted or analyzed outside the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
- c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time, no qualification of the data is necessary.
- d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
- e. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
- f. If technical holding times are grossly exceeded, use professional judgment to qualify the data.

All criteria were metX	
Criteria were not met see below	

GAS CHROMATOGRAPH WITH ELECTRON CAPTURE DETECTOR (GC/ECD) INSTRUMENT PERFORMANCE CHECK (SECTIONS 1 TO 5)

1. Resolution Check Mixture

Criteria

Is the resolution between two adjacent peaks in the Resolution Check Mixture C greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the Yes? or No? confirmation column?

Is the resolution between two adjacent peaks in the Resolution Check Mixture (A and B) greater Yes? or No? than or equal to 60.0%?

Note: If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.

Action

- a. Qualify detects for target compounds that were not adequately resolved as tentatively identified
- b. Qualify non-detected compounds as unusable (R).

2. Performance Evaluation Mixture (PEM) Resolution Criteria

Criteria

Is PEM analysis performed at the required frequency (at the end of each pesticide initial calibration sequence and every 12 hours)? Yes? or No?

Action

a. If PEM is not performed at the required frequency, qualify all associated sample and blank results as unusable (R).

Criteria

Is PEM % Resolution < 90%?

Yes? or No?

Action

- a. a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

All criteria were met	x_
Criteria were not met see below	

3. PEM 4,4'-DDT Breakdown

Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected?

Yes? or No?

Action

a. Qualify detects for 4,4'-DDT; detects for 4,4'-DDD; and detects for 4,4'-DDE as estimated (J)

Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected

Yes? or No?

Action

- a. Qualify non-detects for 4,4'- DDT as unusable (R)
- b. Qualify detects for 4,4'-DDD as tentatively identified (NJ)
- c. Qualify detects for 4,4'-DDE as tentatively identified (NJ)

4. PEM Endrin Breakdown

Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is detected?

Yes? or No?

Action

a. Qualify detects for Endrin; detects for Endrin aldehyde; and detects for Endrin ketone as estimated (J)

Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is not detected

Yes? or No?

Action

- a. Qualify non-detects for Endrin as unusable (R)
- b. Qualify detects for Endrin aldehyde as tentatively identified (NJ)
- c. Qualify detects for Endrin ketone as tentatively identified (NJ)

All criteria were met	X
Criteria were not met see below	20

5. Mid-point Individual Standard Mixture Resolution -

Criteria

Is the resolution between two adjacent peaks in the Resolution Check Mixture C greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column?

Yes? or No?

Is the resolution between two adjacent peaks in the Resolution Check Mixture (A and B) greater than or equal to 90.0%?

Yes? or No?

Note: If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if

coelution exists.

Action

- a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

Criteria

Is mid-point individual standard mixture analysis performed at the required frequency (every 12 hours)?

Yes? or No?

Action

a. If the mid-point individual standard mixture analysis is not performed at the required frequency, qualify all associated sample and blank results as unusable (R).

All criteria were met _	X
Criteria were not met	
and/or see below	

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	12/08/16
Dates of initial calibration verification:	12/08/16
Dates of continuing calibration:	12/14/16
Dates of final calibration	
Instrument ID numbers:	GC1G
Matrix/Level:	Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initia	l and init	ial calib	ration verification within	the quidance docu	ment performance criteria.
		libration	% differences meet the	performance criter	ia in the two columns. Final
			calibration verification in	ncluded in data pac	kage.

Criteria

Are a five point calibration curve delivered with concentration levels as shown in Table 3 of SOP HW-36A, Revision 0, June, 2015?

Yes? or No?

Actions

If the standard concentrations listed in Table 3 are not used, use professional judgment to evaluate the effect on the data

Criteria

Are RT Windows calculated correctly?

Yes? or No?

Action

Recalculate the windows and use the corrected values for all evaluations.

Criteria

Are the Percent Relative Standard Deviation (%RSD) of the CFs for each of the single component target compounds less than or equal to 20.0%, except for alpha-BHC and delta-BHC?

Yes? or No?

All criteria were met _	_X_	
Criteria were not met		
and/or see below		

Are the %RSD of the CFs for alpha-BHC and delta-BHC less than or equal to 25.0%. Yes? or No?

Is the %RSD of the CFs for each of the Toxaphene peaks must be < 30% when 5-point ICAL is performed?

Yes? or No?

Is the %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) less than or equal to 30.0%.

Yes? or No?

Action

- a. If the %RSD criteria are not met, qualify detects as estimated (J) and use professional judgment to qualify non-detected target compounds.
- b. If the %RSD criteria are within allowable limits, no qualification of the data is necessary

Continuing Calibration Checks

Criteria

Is the continuing calibration standard analyzed at the acceptable time intervals? Yes? or No?

Action

- a. If more than 14 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C), qualify all data as unusable (R).
- b. If more than 12 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence, qualify all data as unusable (R).
- c. If more than 72 hours has elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3), qualify all data as unusable (R).

Criteria

Is the Percent Difference (%D) within ±25.0% for the PEM sample?

Yes? or No?

Action

a. Qualify associated detects as estimated (J) and non-detects as estimated (UJ).

Criteria

For the Calibration Verification Standard (CS3); is the Percent Difference (%D) within ± 25.0%? Yes? or No?

Action

Qualify associated detects as estimated (J) and non-detects as estimated (UJ).

Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected?

Yes? or No?

Action

- a. Qualify detects for 4,4'-DDT; detects for 4,4'-DDD; and detects for 4,4'-DDE as estimated (J)
- b. Non-detected associated compounds are not qualified

Criteria

is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected

Yes? or No?

Action

- a. Qualify non-detects for 4,4'- DDT as unusable (R)
- b. Qualify detects for 4,4'-DDD as tentatively identified (NJ)
- c. Qualify detects for 4,4'-DDE as tentatively identified (NJ)

Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is detected?

Yes? or No?

Action

- a. Qualify detects for Endrin; detects for Endrin aldehyde; and detects for Endrin ketone as estimated (J)
- b. Non-detected associated compounds are not qualified

Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is not detected

Yes? or No?

Action

- a. Qualify non-detects for Endrin as unusable (R)
- b. Qualify detects for Endrin aldehyde as tentatively identified (NJ)
- c. Qualify detects for Endrin ketone as tentatively identified (NJ)

All criteria were met	Х_	
Criteria were not met		
and/or see below		

BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contami	ination in the bla	anks below. Hig	h and low levels blanks	must be treated separately.
CRQL concentr	ationN	/A		
Laboratory blan	ks			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_ug/L				nit_of_0.01,_0.02,_and_0.25
Field/Equipme		LEVEL/	COMPOUND	CONCENTRATION
ANALYZED	LAB ID	MATRIX	COMPOUND	UNITS
_Field/equipme	nt_blanks_valida	ated_in_anothe 	r_job	ed_with_this_data_package

All criteria were met __X__ Criteria were not met and/or see below ____

BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

The concentration of non-target compounds in all blanks must be less than or equal to 10 μ g/L. The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method.

Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

Specific actions are as follows:

Blank Actions for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
Method, Sulfur		< CRQL	Report CRQL value with a U
Cleanup, Instrument, Field, TCLP/SPLP	ent, Field,	≥ CRQL and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL and > blank concentration	No qualification required
	= CRQL	≤CRQL	Report CRQL value with a U
		> CRQL	No qualification required
	Gross contamination	Detects	Report blank value for sample concentration with a U

All criteria were met __X__ Criteria were not met and/or see below ____

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES

All criteria were met __X__ Criteria were not met and/or see below_____

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

b

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix:_Aqueou	s			-	
Lab	Lab				
Sample ID	File ID	S1 a	S1 b	S2 a	S2
JC33375-1	1G130505.D	97	90	60	60
JC33375-2	1G130506.D	90	93	77	85
OP99172-BS1	1G130501.D	86	85	45	45
OP99172-MB1	1G130500.D	90	91	42	42
OP99172-MS	1G130503.D	87	88	52	54
OP99172-MSD	1G130504.D	79	77	46	48
Surrogate Comp	pounds		Recove	ery Limit	ts
S1 = Tetrachlor		26-132	%		
S2 = Decachlor		10-118			
(a) Recovery from (b) Recovery from (c)	om GC signal #1 om GC signal #2				

Note: Surrogate recoveries within laboratory control limits.

Actions:

- a. For any surrogate recovery greater than 150%, qualify detected target compounds as biased high (J+).
- b. Do not qualify non-detected target compounds for surrogate recovery > 150 %.
- c. If both surrogate recoveries are greater than or equal to 30% and less than or equal to 150%, no qualification of the data is necessary.
- d. For any surrogate recovery greater than or equal to 10% and less than 30%, qualify detected target compounds as biased low (J-).
- e. For any surrogate recovery greater than or equal to 10% and less than 30%, qualify non-detected target compounds as approximated (UJ).
- f. If low surrogate recoveries are from sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor:
 - i. Qualify detected target compounds as biased low (J-).
 - ii. Qualify non-detected target compounds as unusable (R).

- g. If surrogate RTs in PEMs, Individual Standard Mixtures, samples, and blanks are outside of the RT Windows, the reviewer must use professional judgment to qualify data.
- h. If surrogate RTs are within RT windows, no qualification of the data is necessary.
- i. If the two surrogates were not added to all samples, MS/MSDs, standards, LCSs, and blanks, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

Summary Surrogate Actions for Pesticide Analyses

	Action*			
Criteria	Detected Target	Non-detected Target		
	Compounds	Compounds		
%R > 150%	J+	No qualification		
30% < %R < 150%	No qualification			
10% < %R < 30%	J-	UJ		
%R < 10% (sample dilution not a factor)	J-	R		
%R < 10% (sample dilution is a factor)	Use professional judgment			
RT out of RT window	Use professional judgment			
RT within RT window	No qua	alification		

^{*} Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

All criteria were met	_X	
Criteria were not met		
and/or see below		

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

Data for MS and MSDs will not be present unless requested by the Region.

Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

NOTE: For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:JC33175-1MS/MSD					Matrix/	Level:_	Groundwater		
The QC reported here applies to the following samples: JC33375-1, JC33375-2					Metho	d: SW846 8081B			
	JC33175-1	Spike	MS	MS	Spike	MSD	MSD	RPD	Limits
Dieldrin	ND	0.525	0.54	103	0.525	0.49	93	10	42-161/36

Note: MS/MSD sample analyzed with this data package. % recoveries and RPD within laboratory control limits.

Action

No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

A separate worksheet should be used for each MS/MSD pair.

All criteria were met _	_X_	
Criteria were not met		
and/or see below		

LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

LCS Recoveries Criteria

LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 – 120
Heptachlor epoxide	50 – 150
Dieldrin	30 – 130
4,4'-DDE	50 – 150
Endrin	50 – 120
Endosulfan sulfate	50 – 120
trans-Chlordane	30 – 130
Tetrachloro-m-xylene (surrogate)	30 – 150
Decachlorobiphenyl (surrogate)	30 – 150

%_re	ecovery_a	nd_RPD_within_laboratory_	_control_limits	
	S ID	COMPOUND	% R	QC LIMIT
st the %R of com	pounds w	hich do not meet the criteria	a e	

Action

The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

- a. If the LCS recovery exceeds the upper acceptance limit, qualify detected target compounds as estimated (J). Do not qualify non-detected target compounds.
- b. If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as estimated (J) and non-detects as unusable (R).
- c. Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS.
- d. Use professional judgment to qualify non-LCS compounds. Take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in the performance of the LCS compound to the non-LCS compound.
- e. If the LCS recovery is within allowable limits, no qualification of the data is necessary.

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

All criteria were met	
Criteria were not met	
and/or see belowN/A	

FLORISIL CARTRIDGE PERFORMANCE CHECK

NOTE: Florisil cartridge cleanup is mandatory for all extracts.

Criteria

Is the Florisil cartridge performance check conducted at least once on each lot of cartridges used for sample cleanup or every 6 months, whichever is most frequent?

Yes? or No?

N/A

Criteria

Are the results for the Florisil Cartridge Performance Check solution included with the data package?

Yes? or No?

N/A

Note: If % criteria are not met, examine the raw data for the presence of polar interferences and use professional judgment in qualifying the data as follows:

Action:

- a. If the Percent Recovery is greater than 120% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
- b. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
- c. If the Percent Recovery is greater than or equal to 10% and less than 80% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
- d. If the Percent Recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J) and qualify non-detected target compounds as unusable (R).
- e. If the Percent Recovery of 2,4,5-trichlorophenol in the Florisil Cartridge Performance Check is greater than or equal to 5%, use professional judgment to qualify detected and non-detected target compounds, considering interference on the sample chromatogram.

Note: State in the Data Review Narrative potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results.

Note: No information for florisil cartridge performance check included in data package. There is evidence tahtFlorisil cartridge was used for sample extraction/clean-up. No qualification of the data performed, professional judgment.

All criteria were met_	_N/A
Criteria were not met	
and/or see below	

GEL PERMEATION CHROMATOGRAPHY (GPC) PERFORMANCE CHECK

NOTE: GPC cleanup is mandatory for all soil samples.

If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants; examine subsequent sample data for unusual peaks; and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory chooses to analyze samples under unacceptable GPC criteria.

Action:

- a. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, the non-detected target compounds may be suspect, qualify detected compounds as estimated (J).
- b. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, qualify all non-detected target compounds as unusable (R).
- c. If the Percent Recovery is greater than or equal to 10% and is less than 80% for any of the pesticide target compounds in the GPC calibration, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
- d. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
- e. If high recoveries (i.e., greater than 120%) were obtained for the pesticides and surrogates during the GPC calibration check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.

Note: State in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

Note: No information for performance of GPC cleanup included in data package. No qualification of the data performed, professional judgment.

All criteria were met _	_X
Criteria were not met	1000
and/or see below	

TARGET COMPOUND IDENTIFICATION

Criteria:

- 1. Is Retention Times (RTs) of both of the surrogates and reported target compounds in each sample within the calculated RT Windows on both columns?

 Yes? or No?
- 2. Is the Tetrachloro-m-xylene (TCX) RT ±0.05 minutes of the Mean RT (RT) determined from the initial calibration and Decachlorobiphenyl (DCB) within ±0.10 minutes of the RT determined from the initial calibration?

 Yes? or No?
- 3. Is the Percent Difference (%D) for the detected mean concentrations of a pesticide target compound between the two Gas Chromatograph (GC) columns within the inclusive range of ± 25.0 %?

 Yes? or No?
- 4. When no analytes are identified in a sample; are the chromatograms from the analyses of the sample extract and the low-point standard of the initial calibration associated with those analyses on the same scaling factor?

 Yes? or No?
- 5. Does the chromatograms display the Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.

 Yes? or No?
- 6. If an extract is diluted; does the chromatogram display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale? Yes? or No? N/A
- 7. For any sample; does the baseline of the chromatogram return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB?

 Yes? or No?
- 8. If a chromatogram is replotted electronically to meet these requirements; is the scaling factor used displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram submitted in the data package.

 Yes? or No?

Action:

- a. If the qualitative criteria for both columns were not met, all target compounds that are reported as detected should be considered non-detected.
- b. Use professional judgment to assign an appropriate quantitation limit using the following guidance:
 - If the detected target compound peak was sufficiently outside the pesticide RT Window, the reported values may be a false positive and should be replaced with the sample Contract Required Quantitation Limits (CRQL) value.

- ii. If the detected target compound peak poses an interference with potential detection of another target peak, the reported value should be considered and qualified as unusable (R).
- c. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound should be included.

Note: State in the Data Review Narrative all conclusions made regarding target compound identification.

- d. If the Toxaphene peak RT windows determined from the calibration overlap with SCPs or chromatographic interferences, use professional judgment to qualify the data.
- e. If target compounds were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, use professional judgment to determine how best to report, and if necessary, qualify the data according to these guidelines.
- f. If Toxaphene exhibits a marginal pattern-matching quality, use professional judgment to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of Toxaphene is strongly suggested, report results as presumptively present (N).

GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) CONFIRMATION

NOTE: This confirmation is not usually provided by the laboratory. In cases where it is provided, use professional judgment to determine if data qualified with "C" can be salvaged if it was previously qualified as unusable (R).

Action:

- a. If the quantitative criteria for both columns were met (≥ 5.0 ng/ μ L for SCPs and ≥ 125 ng/ μ L for Toxaphene), determine whether GC/MS confirmation was performed. If it was performed, qualify the data using the following quidance:
 - i. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".
 - ii. If GC/MS confirmation was performed, but unsuccessful for a target compound detected by GC/ECD analysis, qualify those detects as "X".

All criteria were met _	_X_	_
Criteria were not met	.000	
and/or see below		

RF = 0.816

COMPOUND QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JC33375-1 tetrachloro-m-xylene $[] = (146.9 \times 10^{6})(50)/(232.3 \times 10^{6})(0.816)$

Ok

38.7 ppb

Action:

- a. If sample quantitation is different from the reported value, qualify result as unusable (R).
- b. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample.
- c. Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original reporting form and substituting the data from the diluted sample.
- d. Results between the MDL and CRQL should be qualified as estimated (J).
- e. Results less than the MDL should be reported at the CRQL and qualified (U). MDLs themselves are not reported.
- f. For non-aqueous samples, if the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table).

Percent Moisture Actions for Pesticide Analysis for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 < % Moisture < 90.0	J	UJ
% Moisture > 90.0	J	R

DATA REVIEW WORKSHEETS

ampies which h	ave ≤ 50 % solids		
		 	

Note: If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

Dilution performed

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
	· ·	
3.72		
		0.422

All criteria were met_	_N/A
Criteria were not met	
and/or see below	

FIELD DUPLICATE PRECISION

NOTE: In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples. Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. If large RPDs (> 50%) is observed, confirm identification of samples and note difference in the executive summary.

Sample IDs: Matrix:							
COMPOUND	SQL ug/L	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION		
	ug/L	00110.	00140.				
			s data package. MS/M in the required criteria				

Actions:

- a. Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.
- b. If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:
 - i. If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).
 - ii. If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.
 - iii. If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.
 - iv. If both sample and duplicate results are not detected, no action is needed.

OVERALL ASSESSMENT OF DATA Action:

- 1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
- 2. Write a brief narrative to give the user an indication of the analytical limitations of the data.

Note: The Contract Laboratory Program Project Officer (CLP PO) must be informed if any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

Overall assessment of the data: Results are valid; the data can be used for

decision making purposes.

MEMORANDUM

TO: Mr. Haley Royer

Anderson, Mulholland and Associates

DATE: January 14, 2017

FROM: R. Infante

FILE: JC33375

RE: Data Validation

BMSMC, Building 5 Area

SM04.00.06 / 4th Quarter 2016 Groundwater Sampling - Offsite

Accutest Job Numbers: JC33375

SUMMARY

Full validation was performed on the data for two groundwater samples analyzed selected inorganics (iron - ferric and ferrous; nitate-nitrogen; nitrite-nitrogen; nitrate + nitrite - nitrogen; sulfate and sulfide). The methods employed are listed in Table 1. The samples were collected at the BMSMC, Building 5 Area, Humaco, PR site on December 6 2016 and submitted to Accutest Laboratories of Dayton, New Jersey that analyzed and reported the results under delivery groups (SDG) JC33375.

Table 1.

ANALYTE	METHOD	ANALYTE	METHOD		
Iron, ferric	SM3500FE B-11	Iron, ferrous	SM3500FE B-11		
Nitrogen, nitrate ^c	EPA353.2/SM4500NO2B	Nitrogen, nitrate + nitrite	EPA352.2/LACHAT		
Nitrogen, nitrite	SM4500NO2 B-11	Sulfate	EPA 300/SW846-9056A		
Sulfide	SM4500S2-F-11				

- (a) Calculated as: (Iron) (Iron, Ferrous)
- (b) Field analysis required. Received out of hold time and analyzed by request.
- (c) Calculated as: (Nitrogen, Nitrate + Nitrite) (Nitrogen, Nitrite)

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: USEPA Contract Laboratory program National Functional Guidelines for Inorganic data Review (OSWER 9240.1-45, EPA 540-R-04-004, October 2004- Final), (noted herein as the "primary guidance document"). Also, QC criteria from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update IV, December 1998)," and the QC requirements for the methods performed following the Standard Method guidelines are utilized. The guidelines were modified to accommodate the non-CLP methodology. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document. unless otherwise noted.

In general the data are valid as reported and may be used for decision making purposes. The data results are acceptable for use; some of the results were qualified. Results for ferrous and ferric iron were qualified as estimated (J) in samples: JC33375-1 and -2.

SAMPLES

The samples included in the review are listed below

FIELD SAMPLE ID	LABORATORY ID	ANALYSIS
OSMW-3S	JC33375-1	See Table 1
OSMW-4S	JC33375-2	See Table 1

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Surrogate spike recovery
- o Matrix spike/matrix spike duplicate (MS/MSD) results
- o Internal standard performance
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- o Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

Holding Times and Sample Preservation

The cooler temperatures were within the QC acceptance criteria of $4^{\circ}C \pm 2^{\circ}C$.

Sample preservation was acceptable.

Samples analyzed within method recommended holding time except for the following:

- JC33375-1 for Iron, Ferrous: Field analysis required. Received out of hold time and analyzed by request.
- JC33375-2 for Iron, Ferrous: Field analysis required. Received out of hold time and analyzed by request.

Note: Results for ferrous and ferric iron qualified as estimated (J).

Initial and Continuing Calibrations

Initial and continuing calibration meets method performance criteria.

Method Blank/Equipment Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

No field/equipment blanks analyzed as part of this data package.

MS/MSD

Matrix spike was performed. Recoveries for MS/MSD were within laboratory control limits; RPD for MS/MSD were within control limits.

Field/Laboratory Duplicate Results

Field/laboratory duplicate were analyzed as part of this data set. When no field/laboratory duplicates were analyzed, MS/MSD RPD was used to assess precision. RPD results were within laboratory/recommended control limits except for the following:

- JC33258-1/-1 DUP.: Iron, ferrous- 22.2 % RPD, outside laboratory control limit. No action taken, professional judgment. RPD within generally acceptable control limits.
- JC33375-1/-1 DUP.: Nitrogen, nitrate + nitrite- 60.2 % RPD, outside laboratory control limit. No action taken, professional judgment. Sample and duplicate concentration < 5 x IDL.

LCS/LCSD Results

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

Quantitation Limits and Sample Results

Dilutions were not required with this data set.

Calculations were spot checked.

<u>Summary</u>

The following samples JC33375-1 and JC33375-2 were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. Some of the results were qualified, the results are valid.

Rafae Infante

Chemist License 1888

SAMPLE INORGANIC DATA SAMPLE SUMMARY

Sample ID: JC33375-1

Sample location: BMSMC Building 5 Area

Sampling date: 12/6/16

Matrix: Groundwater

Analyte Name	Method	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Fe	SW846-6010C	3020	ug/l	1.0	-	-	Yes
Mn	SW846-6010C	379	ug/l	1.0	(2)	_	Yes
Alkalinity, Total as CaCO3	SM2320 B-11	214	mg/l	1.0	-	~	Yes
Iron, ferric	SM3500FE B-11	3.0	mg/l	1.0		O J.	Yes
Iron, ferrous	SM3500FE B-11	< 0.20	mg/l	1.0		UJ	Yes
Nitrogen, nitrate	EPA 353.2/SM4500NO2B	< 0.11	mg/l	1.0	-	U	Yes
Nitrogen, nitrate + nitrite	EPA 353.2/LACHAT	< 0.10	mg/l	1.0	-	U	Yes
Nitrogen, nitrite	SM4500NO2 B-11	< 0.010	mg/l	1.0	-	U	Yes
Sulfate	EPA 300/SW846 9056A	22.1	mg/l	1.0	-	-	Yes
Sulfide	SM4500S2- F-11	< 2.0	mg/l	1.0	-	U	Yes

Sample ID: JC33375-2

Sample location: BMSMC Building 5 Area

Sampling date: 12/6/2016 Matrix: Groundwater

Analyte Name	Method	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Fe	SW846-6010C	3020	ug/l	1.0	23	U	Yes
Mn	SW846-6010C	454	ug/l	1.0	-	-	Yes
Alkalinity, Total as CaCO3	SM2320 B-11	335	mg/l	1.0	21	-	Yes /
Iron, ferric	SM3500FE B-11	2.9	mg/l	1.0	-		Yes 🗸 🐪
Iron, ferrous	SM3500FE B-11	< 0.20	mg/l	1.0	27	UJ	Yes 🗸 /
Nitrogen, nitrate	EPA 353.2/SM4500NO2B	< 0.11	mg/l	1.0	+0	U	Yes
Nitrogen, nitrate + nitrite	EPA 353.2/LACHAT	<0.10	mg/l	1.0	2	U	Yes
Nitrogen, nitrite	SM4500NO2 B-11	< 0.010	mg/l	1.0	5.65	U	Yes
Sulfate	EPA 300/SW846 9056A	< 10	mg/i	1.0	21	U	Yes
Sulfide	SM4500S2- F-11	< 2.0	mg/l	1.0		U	Yes

DATA REVIEW WORKSHEETS

Type of validation	Full:X Limited: EPA Region:2_	Project Number:JC33375 Date:12/06/2016 Date shipped:12/08/16
		ANALYSIS DATA PACKAGE
sulfide, and/or cyan assist the reviewer is serving the needs of validation guidance Section SOP NO. HIL Laboratory program 45, EPA 540-R-04-Program (CLP) (SO validation criteria we Methods SW-846 (Information (if available))	ide were created to deline in using professional judgrathe data users. The samp documents in the following N-3b Revision 0 (July 2018) National Functional Guide 1004, October 2004- Final DP HW-2, Revision 13. Evere derived from "Test Mether Final Update IV, 1998)". The QC criteria and	als analyses (6010C/6020/7000A series method) ate required validation actions. This document will nent to make more informed decision and in better le results were assessed according to USEPA data grorder of precedence: Hazardous Waste Support of ISM02 ICP-MS Data Validation; USEPA Contractelines for Inorganic data Review (OSWER 9240.1-1). Validation of Metal for the Contract Laboratory assed on ILM05.3 (August 2009). Quality contropods for Evaluating Solid Waste, Physical/Chemical The project QAPP is reviewed for project specifical data validation actions listed on the data review ument, unless otherwise noted.
The hardcopied (la reviewed and the inorganic included:	aboratory name) _Accute quality control and perfo	st data package received has been rmance data summarized. The data review for
No. of Samples: Field blank No.: Equipment blank No	o.:JC33375 2 .:	
X Data deliveX Holding TinX CalibrationX BlanksX ICP InterfeX Matrix Spil	nes	X Laboratory DuplicatesX Field DuplicatesX Laboratory Control SamplesX ICP Serial Dilution ResultsX Detection Limits ResultsX Sample Quantitation
Overall Comments:	_Fe_and_Mn_(SW846-601	10C)
Definition of Qualifie	rs;	.51.52
J- Estimated re U- Compound r R- Rejected da UJ- Estimated no E- Laboratory q	not detected ta on-detect	
Paulawari Ra	rfael Defaut	Dete: 04/4/2017

1 1

				Criteria were not met and/or see below
l.	DATA	DELIVERABLES	3	
	A.	Data Package:		
MISSIN	NG INFO	<u>ORMATION</u>	DATE LAB. CONTACTED	DATE RECEIVED
	1.086-004	-		
	7			
<u> </u>				
	В.	Other Discrepa	ancies:	
				<u> </u>
<u>. </u>				
	docks. —			
	17.2493	10.0000		
	*			-
			6/5/8755-07	
_				
			2030	A 10 1 100 X 10 M
			1100 BBB - BB	

All criteria were met __X_

All criteria were metX
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of preparation, and subsequently from the time of preparation to the time of analysis.

Complete table for all samples and circle the analysis date for samples not within criteria

SAMPLE ID	DATE SAMPLED	CYANIDE DATE ANALYSIS	Hg DATE ANALYSIS	OTHERS DATE ANALYSIS	ρН	SULFIDE	ACTION
SAMPLES	DIGESTED AN	ID ANALYZE	D WITHIN T	HE METHO	D REC	OMMENDE	ED HOLDING
		-					

<u>Criteria</u>

Metals – 180 days from time of collection.
Mercury – 28 days from time of collection.
Hexavalent Chromium (solids)- 30/7 from day of collection; 48 hrs aqueous samples
Cyanide – 14 days from time of collection
Sulfide - 14 days from time of collection
pH measurements of aqueous samples upon receipt at the laboratory (criteria pH ≤ 2 for metals
nH > 12 for cyanide)

Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and rejects nondetects (R)
If pH > 2 for metals or pH < 12 for cyanide, positive results (J) and nondetects (UJ).
Cooler Temperature (Criteria: 4°C + 2°C):5.2°C
If cooler temperature is > 10°C, flag non-detects as (UJ) and detects as (J).

All criteria were metN/A_	
Criteria were not met	
and/or see below	

ICP-MS TUNE ANALYSIS

Is the ICP-MS tuned prior to calibration?

Yes or No?

Does the % RSD exceeds 5% for any isotope in the tuning solution?

Yes or No?

Action:

NOTES: For ICP-MS tunes that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

- 1. If the ICP-MS instrument was not tuned prior to calibration, the sample data should be qualified as unusable (R).
- 2. If the tuning solution was not analyzed or scanned at least 5x consecutively or the tuning solution does not contain the required analytes spanning the analytical range, the reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be recorded in the Data Review Narrative and noted for Contract Laboratory Program Project Officer (CLP PO) action.
- 3. If the resolution of the mass calibration is not within 0.1 u for any isotope in the tuning solution, qualify all analyte results that are ≥ Method Detection Limit (MDL) associated with that isotope as estimated (J), and all non-detects associated with that isotope as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.
- 4. If the %RSD exceeds 5% for any isotope in the tuning solution, qualify all sample results that are ≥ MDL associated with that tune as estimated (J), and all non-detects associated with that tune as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.

Table 2. ICP-MS Tune Actions for ICP-MS Analysis

ICP-MS Tune Results	Action for Samples
Tune not performed	Qualify all results as unusable (R)
Tune not performed properly	Use professional judgment
Resolution of mass calibration not within 0.1u	Qualify results that are ≥ MDL as estimated (J)
	Qualify non-detects as estimated (UJ)
% RSD > 5%	Qualify results that are ≥ MDL as estimated (J)
	Qualify non-detects as estimated (UJ)

Note: Analytes (As) analyzed by SW846-6010 – no tuning necessary.

All criteria were met	X
Criteria were not met	
and/or see below	_

INSTRUMENT CALIBRATION (SECTION 1)

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data. Minimum of 2 calibration points for ICP-AES and ICP-MS; 5 points for Hg; and 4 points for cyanide. One initial calibration standard at the CRQL level for cyanide and Hg. If no, write in the non-compliance section of the data review narrative.

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration Verification standards (ICV or CCV).

Acceptance Criteria	ICV %R	CCV %R
Metals by 6010C/6020	100 + 10%	100 + 10%
Mercury/Metals by 7000s	100 + 10%	100 + 20%
Cyanide	100 + 15%	100 + 15%
Sulfide	100 + 15%	100 + 15%

DATE	ICV/CCV#	ANALYTE	%R	ACTION	SAMPLES AFFECTED
INIT	TAL AND CONT	INUING CALIBE	ATION N	 MEET METHOD SPE	DIFIC CRITERIA

ACTIONS: If any analyte does not meet the %R criteria, follow the actions stated below. Qualify five samples on either side of the ICV/CCV out of control limit.

Estimate positive results (J) if: Metals by 6010C/6020 Mercury/Metals by 7000s Cyanide Sulfide	ICV 111 – 125% 111 – 125% 116 – 130% 116 – 130%	CCV 111 – 125% 111 – 135% 116 – 130% 116 – 130%
Estimate positive results and nondetects (U Metals by 6010C/6020 Mercury/Metals by 7000s Cyanide Sulfide	//UJ) if: 75 – 89% 75 – 89% 70 – 84% 70 – 84%	75 - 89% 65 - 79% 70 - 84% 70 - 84%
Reject positive results and nondetects (R) if Metals by 6010C/6020 Mercury/Metals by 7000s Cyanide Sulfide	f: <75%, >125% <75%, >125% <70%, >130% <70%, >130%	<75%, >125% <65%, >135% <70%, >130% <70%, >130%

All criteria were met	X
Criteria were	not met
and/or see belov	v

- III. INSTRUMENT CALIBRATIONS (SECTIONS 2 & 3)
- 2. Analytical Sequence

Did the laboratory use the proper number of standards for calibration as described in the method?

Yes or No.

B. Were calibrations performed at the beginning of each analysis?

Yes or No.

Were calibration verification standards analyzed at the beginning of sample analysis and the proper frequency according to the method?

Yes or No

D. Where the AA correlation coefficients (r) for the calibration curves
 ≥ 0.995? If r < 0.995, estimate positive results and nondetects (J/UJ).
 It is not necessary to qualify results if the laboratory used order regression.

Yes or No

Data quality may be affected if any of the above answer are "no". Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the sample affected.

3. Other Check Standards

Laboratories may analyze an additional check standard after establishing the calibration curve. This standard may contain low level concentrations of target analytes and be analyzed and evaluated by the laboratory similar to a CLP "CRLD" standard (CRI for ICP, CRA for AA, and/or mid-range standard for CN and Sulfide). A 100 ± 20% recovery acceptance limit should be used by the validator to evaluate the standard.

ACTIONS: If any analyte does not meet the %R criteria, follow the action needed below. Qualify 50% of either side of the CRI/CRA out of control limits.

% R		%R < 50%	%R	=	50-	%R	=	121-	%R	>	Affecte	d Range
			79%			150%			150%			
Qualify Positiv	/e/No	ondetects Res	ults									
Metals	þу	R/R	J/UJ			J/A			R/A		<2x CR	I conc.
6010C/6020	_											
Hg/metals	by	R/R	J/UJ			J/A			R/A		<1.5x	CRI
7000s										ļ	conc.	
Cyanide		R/R	J/UJ			J/A			R/A		<1.5x	mid std.
											conc.	
Sulfide		R/R	J/UJ			J/A			R/A		<1.5x	mid std.
											conc.	

CRI is not required for Al, Ba, Ca, Fe, Mg, Na, and K.

NOTE: CRLD standard within laboratory and method specific criteria.

All criteria were met	N/A
Criteria were	not met
and/or see below	

Table 4. Calibration Actions for ICP-MS Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment
	Qualify results that are ≥ MDL as estimated
	(J)
	Qualify non-detects as estimated (UJ)
Not at least one calibration standard at or	Qualify results that are ≥ MDL but < 2x the
below the CRQL for each analyte	CRQL as estimated (J)
	Qualify non-detects as estimated (UJ)
Correlation coefficient < 0.995; %D outside	Qualify results that are ≥ MDL as estimated
±30%; y-intercept ≥ CRQL	(J)
	Qualify non-detects as estimated (UJ)
Correlation coefficient < 0.990	Qualify results that are ≥ MDL as estimated
	(J)
	Qualify non-detects as unusable (R)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as unusable
	(R)
	Qualify all non-detects as unusable (R)
ICV/CCV %R 75-89%	Qualify results that are ≥ MDL as estimated
	low (J-)
	Qualify non-detects as estimated (UJ)
ICV/CCV %R 111-125%	Qualify results that are ≥ MDL as estimated
	high (J+)
ICV/CCV %R > 125%	Qualify results that are ≥ MDL as estimated
	high (J+)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable
	(R)

All criteria were metX Criteria were not met and/or see below
e existence and magnitude of only to blanks associated with roblems with any blanks exist.

IV. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including equipment, field, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in Sections 1 & 2 below. A separate worksheet page should be used for soil and water blanks.

Laboratory blanks		Matrix:Aqueo	eous	
DATE ANALYZED	ICB/CCB#	PREP BLK	ANALYTE	CONCENTRATION UNITS
No_analyte_de	tected_in_metl	nod_blanks_	_above_reporting_limits	
Field/Equipment			Matrix:Aqueo	us
DATE ANALYZED	EQUIPMENT BLANK	T/FIELD	ANALYTE	CONCENTRATION UNITS
			part_of_this_data_package	

Table. Field/Rinsate/Trip Blank Actions for ICP-MS Analysis

Blank Result	Sample Result	Action for Samples
> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
	> CRQL but < Blank Result	Report at level of Blank Result with a "U"
	> Blank Result but < 10x the Blank Result	Use professional judgment to qualify results as estimated (J)

	A	All criteria were metX Criteria were not met and/or see below
IV.	BLANK ANALYSIS RESULTS (Section 3)	
Freque	ncy requirements	
at the fi	e preparation blank analyzed for each matrix, requency of the method? stimate positive results < 10x IDL for which preparation blank w than 20 samples/batch, qualification begins at the 21 st sample.	Yes or No as not analyzed.
B.	Was an ICB analyzed?	Yes or No
C.	Was a CCB analyzed at the frequency stated in the method?	Yes or No
determ	uality may be affected if any of the above answer is "no". Us ine the severity of the effect and qualify the data accordingly. Ithe samples affected.	e professional judgment to Discuss any actions below,
	20	
Compa	FOR SOIL SAMPLES are raw sample value with blank results in ug/L unit, or t blanks analyzed during a soil case to mg/Kg in order to com	pare them with the sample
	n ug/L x [Volume diluted to (mL)]/[Weight digested] x 1L/1000r 000□g = concentration in wet weight (mg/Kg)	mL x 1000g/1Kg x
Concer	ntration, dry weight (mg/Kg) = (Wet weight concentration)/(% So	lids) x 100
BLANK	ANALYSIS RESULTS (Sections 4,5)	
sample	ntamination remaining in the field or equipment blank will be use	

1 1

			All criteria were n Criteria we and/or see be	ere not met
4. Initial/	Continuing Cali	bration Blanks (ICB/C0	CB) Actions	
Are all ICB/CC	Bs less than th	e SQL?	Yes or No	
		either side of the ICB/0 the ICB/CCB value.	CCB out of control limits.	
ICB/CCB#	ANALYTE	CONC/UNITS	SAMPLES AFFECTED	
				_
				<u>-</u>
Are the PB les	ss than the SQL	?	Yes or No	_
lf yes, reject a	II results (R) < 1	0x the PB value.		
РВ	ANALYTE	CONC/UNITS	SAMPLES AFFECTED	
				_
				_
				_
BLANK ANAL	YSIS RESULTS	S (Section 6)		
6. Field/E	Equipment Blan	k (FB/EB) Actions		
Are th	e FB/EB less th	an the SQL?	Yes or No	N/A
if no, was the	FB/EB value alr	eady rejected due to c	other QC criteria? Yes or No	
lf no, reject (R the FB/EB valu		s <_5x the FB/EB value	e. Reject soil data with raw digest r	esults < 5x
PB	ANALYTE	CONC/UNITS	SAMPLES AFFECTED	
				_
			· · · · · · · · · · · · · · · · · · ·	_

All criteria were met	N/A
Criteria were	not met
and/or see below	٧

Table 5. Calibration/Preparation Blank Actions for ICP-MS Analysis - Summary

Blank Type	Blank Result	Sample Result	Action for Samples	
ICB/CCB	≥ MDL but ≤ CRQL	Non-detect	No action	
≥ MDL but ≤ CRQL		Report CRQL value with	a "U"	
> CRQL		Use professional judgme	ent	
ICB/CCB	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"	
> CRQL but < Blank Res	sult	Report at level of Blank	Result with a "U"	
> Blank Result		Use professional judgme	ent	
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment	
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-)	
			Qualify non-detects as estimated (UJ)	
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"	
> CRQL but < 10x the B	lank Result	Qualify results as estimated high (J+)		
≥ 10x the Blank Result		No action		
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action	
≥ MDL but ≤ CRQL		Report CRQL value with a "U"		
> CRQL		Use professional judgment		
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-)	
			Qualify non-detects as estimated (UJ)	

				Crite	vere metX eria were not met see below
INDUCTIVELY CO	OUPLED PLAS	SMA (ICP) INTER	RFERENCE CHEC	CK SAMPLE	
The assessment interelement and			eck sample (ICS)	is to verify	the laboratory's
1. Recovery	Criteria				
List any elements %).	in the ICS AB	and ICS A soluti	ions which did not	meet the %R	criteria (80 – 120
DATE E	LEMENT	%R ACTION	N SAMPLES	SAFFECTED	
_Interference_che	eck_sample_wi	ithin_method_pe	rformance_criteria		
ACTIONS: If an element does	s not meet the	%R criteria, follo	w the actions state	ed below	
% R	%R < 50%	%R = 50- 79%	%R = 121- 150%	%R >	
Qualify Positive/N		ults]
Metals by 6010C/6020	R/R	J/UJ	J/A	R/A	
2. Frequenc	y requirements	3			
Were interference (beginning of the			ncy stated in the m		s or No
If no, ACTIONS: Estima	ate positive res	ults (J) all sample	es for which Al, Ca	a, Fe, Mg > ICS	S value.
The data may be	affected. Use	professional judg	gment to determing below and list the	e the severity	of the effect and
		CTAR			
					See to a consecue

All criteria were metN/A	
Criteria were not me	et
and/or see below	

Table 6. Interference Check Actions for ICP-MS Analysis - Summary

Interference Check Sample Results	Action for Samples
ICS not analyzed	Qualify detects and non-detects as unusable (R)
ICS not analyzed in proper sequence	Use professional judgment.
ICS %R>150%	Use professional judgment
ICS %R > 120% (or greater than true value + 2x the CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 80-12-%	No qualification
ICS %R 50-79% (or less than true value – 2x the CRQL)	Qualify results that are ≥ MDL as estimated low (J-)
	Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify detects as estimated low (J-) and non- detects as unusable (R)
Potential false positives in field samples with interferents	Qualify results that are ≥ MDL as estimated high (J+)
Potential false negatives in field samples with interferents	Qualify results that are ≥ MDL but < 10x the (negative value) as estimated low (J-) Qualify non-detects as estimated (UJ)

	C	were metX Criteria were not met /or see below
/I. MATRIX SPIKE (MS)		
Sample # _JC33148-1MS/-1MSD	Matrix:Groundwater	Units:ug/L

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. Note that for Region 2, MS not required for: Ca, Mg, K, and Na for aqueous matrix.

Al, Ca, Fe, Mg, K, Na, for soil matrix

MS Recovery Criteria. List the percent recoveries for analytes which did not meet the %R criteria (75 – 125%); (85 – 115 % FOR Cr (VI)).

ANALYTE	SPIKE SAMPLE	SAMPLE	SPIKE	% R	ACTION
	RESULT (SSR)	RESULT (SR)	ADDED		
MS/MSD recoveries and RPD within laboratory control limits.					
					\$1.90A.9\$
					-

ACTIONS: Matrix spike actions apply to all samples of the same matrix. The qualification will also be applied to the results of all samples within a given area of the site, if deemed appropriate.

If the sample results \geq 4x the spike concentration, no action is taken. If any analyte does not meet the %R criteria, follow the actions stated below.

Table 9. Spike Sample Actions for ICP-MS Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion Spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-digestion spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)

DATA REVIEW WORKSHEETS

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

2. Frequency Criteria

A. Was a matrix spike prepared at the frequency stated in the method (1/20)? Yes or No

If no, estimate positive results (J) for which analyte was not spiked. If more than 20 samples/batch, qualification begins at the 21st sample.

B. Was a field blank used as spiked sample? Yes or $\underline{\text{No}}$ If yes, estimate positive results (J) < 4x spike level added for the analyte.

A separate worksheet page should be used for each matrix spike

	All crite	eria were metN/A Criteria were not met and/or see below
VII. FIELD DUPLICATES		
Sample #:	Matrix:	Units:_ug/L

Field duplicate samples may be taken and analyzed as an indication of overall precision. Field duplicate analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

List the concentrations and RPDs in the field duplicate pair. RPD criteria: \pm 20% for aqueous; \pm 35% for soil. For soil duplicates, if the % solids for the sample and its duplicate differ by more than 1%, report concentrations in ug/L and calculate RPD or difference for each analyte.

ANALYTE	SQL ug/L	SQL ug/Kg	SAMPLE RESULTS	DUPLICATE RESULTS	RPD	ACTION
Al	ug/L	ug/itg	RESOLIS	INEGUETO		
Sb	+	+				
	NI- C-1	10-1	l Protest		1	100.0/
As	used to	o assess p	recision. RPD	naiyzed with da within laborato limits	ry and gene	MSD % recoveries RPD erally acceptable control
Ba						
Be						
Cd	1					
Ca	1					
Сг						
Co						
Cu	1					
Fe						
Pb						
Mg						
Mn						
Hg						
Ni	T					
K	1					
Se						
Ag						
Na						
TI						
V						
Zn						
Cyanide						
Cr(VI)						

Field duplicate actions should be applied to only the sample and its duplicate.

All criteria were n	net	N/A	
Criteria	were	not	met
and/or se	e belo	w	

<u>Actions:</u> Indicates which criterion was used to evaluate precision by circling either the RPD or SQL for each element. If both sample and duplicate are nondetects, the RPD is not calculated (NC), no action is needed.

Table 8. Duplicate Sample Actions for ICP-MS Analysis

Duplicate Sample Results	Action for Samples
Aqueous: Both original sample and duplicate sample > 5x the CRQL and 20% < RPD < 100%	Qualify those results that are ≥ CRQL as estimated (J)
Aqueous: Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 100%	Qualify those results that are ≥ CRQL as unusable (R)
Soil/Sediment: Both original sample and duplicate sample > 5x the CRQL and 35% < RPD < 120%	Qualify those results that are ≥ CRQL as estimated (J)
Soil/Sediment: Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 120%	Qualify those results that are ≥ CRQL as unusable (R)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL	Qualify those results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ)

A separate worksheet page should be used for each laboratory duplicate analysis

				Criteria were not met and/or see below
VIII. LABOR	ATORY DUPLICATES (Section 1)		
measure of lab	duplicates samples to poratory performance. It the than water matrices diles.	is also expe	cted that soil duplic	ate results will have a
1. Difference Co	iteria			
for soil). For so	trations of any analyte n il duplicates, if the % so centrations in □g/L and ca	olids for the s	ample and its duplic	ate differ by more than
Sample #		Matrix:	<u></u>	Units:

ANALYTE	SQL ug/L	SQL mg/Kg	SAMPLE RESULTS	DUPLICATE RESULTS	RPD	ACTION
Al						
Sb						
As						
Ва						
Be						
Çd						
Ca						
Cr						
Co						
Cu						
Fe	-					
Pb						
Mg						
Mn	T					
Hg						
Ni						
K						
Se						
Ag						
Na						
TI						
V						
Zn						
Cr(VI)						
Sulfide						
Cyanide	- 123					

Note:

Laboratory duplicates actions should be applied to all other samples of the same matrix type. This qualification will also be applied to the results of all samples within a given area of the site, if deemed appropriate.

All criteria were met __N/A___ Criteria were not met and/or see below ____

Actions: Indicates which criterion was used to evaluate precision by circling either the RPD or SQL for each element. If both sample and duplicate are non-detects, the RPD is not calculated (NC), no action is needed.

Table 8. Field Duplicate Sample Actions for ICP-MS Analysis

Sample Type	Field Duplicate Result	Action for Samples
Aqueous	Sample and its field duplicate ≥ 5x the CRQL and RPD > 20%	Qualify sample and its duplicate as estimated (J)
	Sample and/or its field duplicate < 5x the CRQL and absolute difference > the CRQL	Qualify results > the MDL as estimated (J) Qualify non-detects as estimated (UJ)
Soil/Sediment	Sample and its field duplicate ≥ 5x the CRQL and RPD > 50%	Qualify sample and its duplicate as estimated (J)
	Sample and/or its field duplicate < 5x the CRQL and absolute difference > 2x the CRQL	Qualify results > the MDL as estimated (J)
		Qualify non-detects as estimated (UJ)

2. Frequency Criteria

A. Was a laboratory duplicate prepared at the frequency stated in the method (1/20)? Yes or No

If no, estimate positive results (J) for the analyte which duplicate was not performed. If more than 20 samples/batch, qualification begins at the 21st sample.

B. Was a field blank used for laboratory duplicate analysis? Yes or No

If yes, estimate positive results (J) for the analyte if field blank was used for duplicate analysis.

All criteria were metX
Criteria were not met
and/or see below

IX. LABORATORY CONTROL SAMPLE (LCS/LCSD)

The assessment of the LCSs is to determine both intralaboratory contamination and matrix specific precision and accuracy. Note that for Region 2, LCS is not required for aqueous Hg and Cyanide.

LCS Recoveries Criteria

A. <u>Aqueous LCS</u>/Solid LCS

List any LCS recoveries not within %R criteria (80 – 120%) and the samples affected.

DATE	ELEMENT	% R	ACTION	SAMPLES AFFECTED
 Recoveries_w	vithin_laboratory_control	_limits		

ACTIONS: If analyte does not meet the %R criteria, follow the actions stated below:

Table 7. LCS Actions for ICP-MS Analysis

LCS Result	Action for Samples
%R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
%R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
%R 70-130%	No qualification
%R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
%R > 150%	Qualify detects as unusable (R); non- detects no qualification

All criteria were metX
Criteria were not met
and/or see below

2. Frequency Criteria

A. Was a laboratory control sample prepared at the frequency stated in the method (1/20)? **Yes** or No

If no, estimate positive results (J) for the analyte if LCS was not performed.

If more than 20 samples/batch, qualification begins at the 21st sample.

							,	Cr	iteria we	etX_ re not met low
X.	ICP SER	IAL DIL	UTION AN	NALYSIS (S	ection 1)					
	sessment ı a 5x dilut		CP serial	dilution ana	lysis is to	deter	rmine the	e precisio	n of the	laboratory
1.	Percent (Differen	ce (%D) C	riteria:						
	s analysis			erformed 1 0% of the u						
	Serial	dilutions	were	not perf	ormed	for	the fo	llowing	target	analytes:
	lyte conce	ntration	s > 50x ID	rmed, but ar L before dil	ution.				hin 10%	difference
		•				·		,		
Sample	# _ JC33	3148-1_			Matrix	:Gro	oundwate	er	Units:_	_ug/L
ANALY	TE	IDL	50x IDL	SAMPLE	SERIA	۱L	%D	ACTION	1	

ANALYTE	IDL	50x IDL	SAMPLE RESULTS	SERIAL DILUTION	%D	ACTION
Al						
Sb						
As						
Ва					T	
Ве						
Cd						
Ca						
Сг						
Со					1	
Cu		İ	İ			
Fe					1	
Pb						
Mg						
Mn						
Hg						
Ni		1				
K						
Se						
Ag						
Na						
TI						
V						
Zn					1	İ

Note: Serial dilution within method performance criteria.

All criteria were metX
Criteria were not met
and/or see below

ACTIONS: Actions apply to all samples of the same matrix. The qualification will also be applied to the results of all samples within a given area of the site, if deemed appropriate. Qualify only samples with raw results > 50x MDL.

Flag results with an (E) for elements exhibiting %D > 10%. Estimate (J) positive results > 50x MDL for elements that exhibited %D > 10 but < 100.

Reject (R) positive results > 50x MDL for elements which exhibited %D ≥ 100 %.

SERIAL DILUTION ANALYSIS (Section 2)

2. Frequency Criteria

A. Was a serial dilution analysis prepared as required by the method? Yes or No

If no, estimate positive results \geq 50x MDL (J) for the analyte which serial dilution analysis was not performed.

B. Was a field blank used for serial dilution analysis?

Yes or No

If yes, estimate positive results \geq 50x MDL (J) for the analyte if field blank was used for serial dilution analysis.

Table 10. Serial Dilution Actions for ICP-MS Analysis

Serial Dilution Result	Action for Samples
Aqueous: Sample concentration > 50x MDL and 10% < %D < 100%	Qualify affected results whose raw data are > MDL as estimated (J)
Aqueous: Sample concentration > 50x MDL and %D ≥ 100%	Qualify affected results whose raw data are > MDL as unusable (R)
Soil/Sediment: Sample concentration > 50x MDL and 15% < %D < 120%	Qualify affected results whose raw data are > MDL as estimated (J)
Soil/Sediment: Sample concentration > 50x MDL and %D ≥ 120%	Qualify affected results whose raw data are > MDL as unusable (R)
Interferences present	Use professional judgment

A separate worksheet page should be used for each serial dilution analysis.

Criteria were not met d/or see below	
	. ICP-MS INTERNAL STANDARDS
Yes_or No?	Are internal standard added to the sample?
Yes or No?	Are the proper number of internal standard added to the sample?
within 60-125% of the Yes or No?	Is the % Relative Intensities for all internal standards in a sample response in the calibration blank?
in_the_guidance_	Note:_ICP-OES_internal_standards_used;_relative_intensities_w _document_performance_criteria
	document_performance_criteria

All criteria were met N/A

Action:

NOTE: Apply the action to the affected analytes for each sample that does not meet the internal standard criteria.

- 1. If no internal standards were analyzed with the run, the sample data should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP Project Officer (CLP PO) action.
- 2. If less than five of the required internal standards were analyzed with the run, or a target analyte(s) is (are) not associated to an internal standard, the sample data, or analyte data not associated to an internal standard should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP PO action.
- 3. If the % Relative Intensities for all internal standards in a sample is within 60-125% of the response in the calibration blank, the sample data should not be qualified.
- 4. If the %RI for an internal standard in a sample is not within the 60-125% limit, qualify the data for those analytes associated with the internal standard(s) outside the limit as follows:
 - a. If the sample was reanalyzed at a two-fold dilution with internal standard %RI within the limits, report the result of the diluted analysis without qualification. If the %RI of the diluted analysis was not within the 60-125% limit, report the results of the original undiluted analyses and qualify the data for all analytes that are ≥ Method Detection Limit (MDL) in the sample associated with the internal standard as estimated (UJ).
 - b. If the sample was not reanalyzed at a two-fold dilution, the reviewer should use professional judgment to determine the reliability of the data. The reviewer may determine that the results are estimated (J) or unusable (R).

DATA REVIEW WORKSHEETS

Table 11. Internal Standard Actions for ICP-MS Analysis

Internal Standard Results	Action for Samples
No internal standards	Qualify all results as unusable (R)
< 5 of the required internal standards	Qualify all results as unusable (R)
Target analyte not associated with internal standard	Qualify all analyte results not associated with an internal standard as unusable (R)
% RI < 60% or > 125%, original sample	Do not qualify the data
reanalyzed at 2-fold dilution, and % RI of diluted	·
sample analysis is between 60% and 125%	
% RI < 60% or > 125%, original sample	Qualify analytes associated with the failed
reanalyzed at 2-fold dilution, and % RI of diluted	internal standard that are ≥ MDL as estimated
sample analysis is outside the 60% to 125% limit	(J) and qualify associated non-detects as estimated (UJ)
Original sample not reanalyzed at 2-fold dilution	Use professional judgment
A	Qualify sample results as estimated (J) or unusable ®

DATA REVIEW WORKSHEETS

XII. DETECTION LIMITS RESULTS

The detection limit assessment is to verify that samples results are within instrument calibration range or linear range (ICP).

Instrument Detection Limits (IDL). Note IDL is not required for Cyanide.

- A. IDL/MDL (or lowest quantitation limit used) results were present and found to be all levels that meet the project objectives? Yes or No
- B. IDL/MDL (or lowest quantitation limit used) were not met for the following elements:
- 2. Reporting Requirements
- A. Were sample results on Form I (or equivalent) reported down to the IDL/MDL or lowest quantitation limit used for all analytes?

 Yes or No
- B. Were sample weights, volumes, and dilutions taken into account when reporting results (positive and nondetects)? Yes or No

If no, the reported results may be inaccurate. Request the laboratory resubmit the corrected data.

- 3. Sediment Sample Percent Solids (% solids):
- A. Were the % solids for any sediment samples < 50% but ≥ 10%? Yes or No If yes, estimate positive results and nondetects (J/UJ) if the % solids is 10-50%. List the affected samples:_____
- B. Were the % solids for any sediment samples < 10%? Yes or No If yes, reject all results (R) if the % solid is < 10%. List the affected samples: N/A
- XI. TOTAL/DISSOLVED OR INORGANIC/TOTAL ANALYTES
- A. Were any analyses performed for dissolved as well as total analytes on the same sample(s)?

 Yes or No
- B. Were any analyses performed for inorganic as well as total analytes on the same sample(s)?

 Yes or **No**

If yes, compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference as a percent of the total analyte only when both of the following conditions are fulfilled:

- (1) The dissolved (or inorganic) concentration is greater than total concentration, and
- (2) greater than or equal to 5xMDL.

	A	All criteria were metN/A
		Criteria were not met and/or see below
C.	Is any dissolved (or inorganic) concentration greater than it than 20%? Yes or No	s total concentration by more
D.	Is any dissolved (or inorganic) concentration greater than it than 50%? Yes or No	s total concentration by more
	N: percent difference is greater than 20%, flag (J) both c trations as estimated. If the difference is more than 50%, reje	
XII.	SAMPLE QUANTITATION	
The sa	mple quantitation evaluation is to verify laboratory quantitation	results.
	Sample results fall within the linear range for ICP and within arameters.	n the calibration range for all
dilution	If samples results were beyond the linear range/calibration performed?	range of the instrument, were
List the	affected samples/elements/dilution:	
In the s	pace below, please show a minimum of one sample calculation	on per method:
ICP/ICE	Computer printout	
<u>Hq/Met</u>	als by AA	
Hexava	alent Chromium	
Cyanid	<u>e</u>	
<u>Others</u>		
	samples, the following equation may be necessary to conve actual sample concentrations (mg/Kg):	rt raw data values reported in
Conc. i	n ug/L x <u>Volume diluted to, mL</u> x <u>1L</u> x <u>1000 g</u> x <u>1 mg</u> Weight digested, g 1000 mL 1 Kg 100	_ = concentration 00 mg in wet weight mg/Kg
In addit	ion the sample results are converted to dry weight by using the	ne percent solid calculations:

Wet weight concentration x 100 = final concentration, dry weight (mg/Kg) % solids

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DATA REVIEW WORKSHEETS

OVERALL ASSESSMENT

Action:

- 1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.
- 2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the Sample Delivery Group (SDG) Narrative for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
- 3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

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